LABORATORY CHARACTERIZATION OF BLENDS WITH RECYCLED MATERIALS USING HIGH REPLACEMENT BINDER RATIOS AND A RECYCLING AGENT TYPE

A Thesis

by

JUAN SEBASTIÁN CARVAJAL MUÑOZ

Submitted to the Office of Graduate and Professional Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Chair of Committee,	Amy Epps Martin
Committee Members,	Jon Epps
	Charles Glover
Head of Department,	Robin Autenrieth

December 2015

Major Subject: Civil Engineering

ABSTRACT

The effects of using recycling agents (RAs) on hot mixture asphalt (HMA) have recently acquired special attention in the academic community due to the fact that recycled mixtures typically have high stiffness as a result of long periods of oxidative aging, mainly driven by environmental conditions (e.g., sunlight, temperature, and oxygen). In addition, the use of RAs in HMA aims at restoring the rheological and physicochemical properties of the aged binder contained in the reclaimed asphalt pavement (RAP) and reclaimed asphalt shingles (RAS) to provide the HMA mixtures with adequate performance, similar or superior to that of virgin HMA (i.e., without RAP, RAS, or RA). Subsequently, the objective of this thesis is to study the rheological and ageing characteristics of asphalt binder blends fabricated with RAP, RAS, and recycling agents using high recycled binder ratios (RBRs). The materials were retrieved from a recent field project constructed in Texas that incorporated high percentages of RAP/RAS materials with recycling agents and warm mixture asphalt additives in five highway sections. The laboratory characterization included: (1) blending charts, dosage estimations, and master curves from dynamic shear rheometer, (2) the Glover-Rowe (G-R) parameter in Black Space diagrams, and (3) Fourier transform infrared spectroscopy (FTIR).

The results indicated that the use of the T1 recycling agent allowed for restoring the rheology (i.e., G* and phase angle) in all the combinations studied. Furthermore, validation of the regional linear blending concept allowed for calculating dosages of RA.

The G-R parameter in Black Space diagrams showed that more than 40 pressure aging vessel (PAV) hours of artificial aging produce high stiff blends with high cracking susceptibility (i.e., damage zone between 180 kPa and 450 kPa). Furthermore, FTIR data showed a tendency of increase in the carbonyl area when using the T1 RA. Results include a suggested set of laboratory tools for characterizing binder blends, and a proposed methodology for determination of optimum dosages of RA in recycled mixtures with high RBR to be used in future research.

DEDICATION

God is the inspiration for everything I do in my life. His guidance, eternal patience, and infinite love have allowed me to overcome any obstacle in my life. His greatest gifts to me are my beloved parents, who have always given me the strength and motivation to keep going in the right path to become a functional citizen and, more importantly, a better human being every day. My brother, Ricardo, and my sister, Ivette, have always been there for me to give advice to me, reach for me when I am feeling alone, or give me moments of joy and happiness when we are together. Their love accompanies me always and is a great source of my happiness.

I also dedicate this work to my eternally beloved and charismatic wife, Charlie E. Carmona, who has been with me in more recent years, giving me many reasons to be grateful and happy. My family's presence in my life has made everything easier and has allowed me to see life from a different perspective: from the perspective of love, persistence, discipline, humbleness, and positive attitude toward any circumstance faced along the way.

ACKNOWLEDGEMENTS

First, I thank the continuous support provided by my Chair, Dr. Epps Martin, who gave me the confidence and technical knowledge required to become a more consistent, disciplined, and focused researcher. Second, I express my gratitude to Dr. Allex E. Alvarez, who during my undergraduate studies at University of Magdalena taught me the great impact that research can have on people's lives and widened my mind to new opportunities that created virtually limitless expectations for my future. His advice, technical discussions, and role as professor helped to shape the professional and human being I am today.

I also acknowledge the members of the National Cooperative Highway Research Program (NCHRP Project 9-58) for their valuable inputs and insights on the topic of this research, especially the advice from Dr. Charles Glover, Dr. Jon Epps, Dr. Edith Arambula, and Dr. Fujie Zhou. Additional gratitude is expressed to Lorena Garcia, Fan Yin, Shi Chang, Fawaz Kaseer, Tony Barbosa, and Rick Cantanella for their help in many of the experimental aspects of this work and their friendship during my time at Texas A&M University.

NOMENCLATURE

AV	Air Void
BBR	Bending Beam Rheometer
CA	Carbonyl Area
δ	Phase Angle for the Asphalt Binder
DSR	Dynamic Shear Rheometer
FTIR	Fourier Transform Infrared Spectroscopy
G^*	Complex Modulus for the Asphalt Binder
G-R	Glover-Rowe Parameter
HMA	Hot Mixture Asphalt
LSV	Low-Shear Viscosity
LVE	Linear Viscoelastic Range
MWAS	Manufactured Waste Asphalt Shingles
NAPA	National Asphalt Pavement Association
NCHRP	National Cooperative Highway Research Program
PAV	Pressure Aging Vessel
PG	Performance-Graded
PGL	Low-Temperature PG
PGH	High-Temperature PG
RA	Recycling Agent
RAP	Reclaimed Asphalt Pavement

RAS	Recycled Asphalt Shingles
RBR	Recycled Binder Ratio
RTFO	Rolling Thin Film Oven
T1	Tall-Oil Recycling Agent
TCE	Trichloroethylene
TTI	Texas A&M Transportation Institute
TxDOT	Texas Department of Transportation
ZSV	Zero Shear Viscosity

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1. INTRODUCTION

Use of recycled materials in asphalt mixes is now a common practice due to stricter environmental regulations and cost-saving strategies implemented in highway infrastructure projects. In fact, recycled asphalt pavement (RAP) and recycled asphalt shingles (RAS) are currently in use by several state departments of transportation (DOTs) in the United States due to their benefits, which include lower consumption of virgin materials, reduction of storage in landfills, and cost savings with regard to the lower demand of virgin binder required to comply with design guidelines. Nevertheless, some previous studies have reported performance issues such as fatigue and low-temperature cracking when high RAP/RAS ratios are used. The degree of blending occurring between virgin and aged asphalt binder has also been considered a critical aspect with respect to the effectiveness of rejuvenation of the recycled binder and mixture performance over time. Numerous research efforts have been undertaken toward better understanding the mechanical response of hot mixture asphalt (HMA) with high RAP/RAS by the combination of asphalt binder, coarse and fine aggregates, and additives as a whole. However, the individual assessment of the asphalt binder has been limited, especially for experimental designs including high-recycled binder ratio (RBR). RBR is defined as the ratio of recycled binder to the percentage of virgin binder. For instance, a 0.3 RBR indicates a 30 percent contribution from the aged binder, with the remaining 70 percent from the virgin binder. Typically, high RBRs are those above 25 percent addition of aged asphalt binder.

Research on this topic includes National Cooperative Highway Research Program (NCHRP) Project 9-12, which incorporated some recommendations for mixture design with inclusion of RAP following the Superpave™ standards. More recently, NCHRP Project 9-46 devoted more efforts toward mixture design procedures for high RAP mixtures (>25% RAP), subsequently suggesting changes in the current specifications for improved long-term performance. Currently, NCHRP Project 9-58 is ongoing in an attempt to study mixtures with inclusion of recycled materials with high RBRs and recycling agents (RAs) in various dosages to evaluate the overall impact that these have on mixture performance and component material properties.

In particular, RAs are added to the HMA with the purpose of minimizing the impact that the aged binder from RAP/RAS has on the mixture stiffness and also to restore some rheological and physicochemical properties of the asphalt binder blend. Moreover, the effects of using RAs on HMA are of special interest due to the fact that recycled mixtures typically have high stiffness, derived from long periods of oxidative aging with exposure to environmental conditions (e.g., sunlight, temperature, oxygen). For these reasons, recent research has focused on better understanding the effect of RAs on HMA through multiple approaches that include laboratory characterization and field performance assessment. These have also involved a wide range of technologies and equipment developed by the asphalt industry.

Some supporting theory indicates that the use of RAs in HMA aims at restoring the rheological and physicochemical properties of the aged binder contained in the RAP and RAS to provide the HMA mixtures with adequate performance, similar or superior to that of virgin HMA (i.e., with no RAP, RAS, or RAs). In addition, this RA becomes of great relevance when considering the use of high RBR in HMA as a result of environmental regulations and cost savings in HMA production.

Typically, the percentages of addition for recycled materials are restricted to maximum values due to their impact on the overall stiffness of the recycled mixture, which can induce premature distresses in the asphalt pavement layers, such as fatigue cracking and low-temperature cracking. Thus, use of high RBRs has attracted significant attention, translated into a wide variety of research and field projects focused on better understanding the effects of the addition of RAs on the short- and long-term performance of HMA, as well as other theoretical approximations in terms of the materials properties and mechanisms through which rheology and physicochemical properties are restored.

In this context, the blending occurring between the recycled and the virgin materials is not well understood at higher RBR than those conventionally used, which represents both a challenge and an important topic for a more comprehensive characterization of recycled mixture with high RBR (i.e., above 25% replacement for RAP/RAS). Subsequently, the laboratory characterization of blends with high RBR is essential to understand at what level the materials are compatible and how the RAs affect the rheological properties and aging evolution of the recycled mixture.

Furthermore, this type of evaluation is needed for promoting better practices in paving engineering when recycled materials and RAs are considered.

1.1. Research Objectives

Considering the aforementioned, the main objective of this research is to evaluate the effects of RAs on HMA mixtures containing RAP and RAS at high RBRs by laboratory characterization of binder blends. The testing plan characterizes the blends via rheological and chemical assessments (i.e., using the dynamic shear rheometer [DSR] and Fourier transform infrared spectroscopy [FTIR]). These evaluations will allow the optimum dosage of RA to be used to restore the performance grade of the aged binder blend to be determined, as well as provide improved understanding of the effect of these RAs on the rheology and aging processes occurring in the blends with high RBRs.

1.2. Description of Contents

This document is comprised of the following sections: a literature review in section 2 that presents information with regard to recycling materials, recycling agents, blending and aging. This section is followed by the experimental design in section 3 that describes the materials; methods and laboratory tests carried out for characterizing asphalt binders and binder blends.

Test results and data analysis are subsequently presented in section 4. Section 5 focuses on the contribution to research that includes a set of tools for characterizing binder blends and a methodology for determining optimum dosage by characterizing virgin binder, binder blends and asphalt mixtures. Conclusions and recommendations in section 6 complete the thesis, and an appendix is included, which presents the asphalt extraction and recovery protocol.

2. LITERATURE REVIEW

This section provides a summary of the conceptual aspects related to the use of recycled materials in HMA with high RBRs and RAs, with a focus on the asphalt binder. The information is divided into the following major topics:

- Recycling of Asphalt Materials
- Effect of RAs on Binders with High RBRs
- The Concept of Blending
- Aging Characterization

2.1. Recycling of Asphalt Materials

Several materials that have been successfully and widely recycled in pavements include: foundry sand, glass, slags, and tire rubber. However, the most commonly used recycled material in the asphalt industry is RAP. Appropriate RAP management, mixture design, and production considerations are important aspects to consider in the fabrication of HMA that complies with normal specification requirements. However, to achieve that, substantial testing is still required to thoroughly characterize the recycled material and better understand its rheological and physicochemical properties.

The binder contained in RAP is aged to levels that depend upon several factors, such as pavement age, climate type, mixture air void content, and binder grade used in the original pavement. As expected, the aging level of the RAP can have both beneficial and detrimental effects on the mixture performance. At high service temperatures it may improve rutting resistance, but the cracking resistance at intermediate and low temperatures might be compromised due to the high stiffness of the asphalt layers.

Conversely, as a result of the need to save on costs and maximize efficiency, major attention has been directed toward the use of RAS in recycled mixtures because this material has higher asphalt binder content than RAP (typically in the range of 20 to 30 percent by total weight). In general, two major types of RAS are commonly used: manufacturer waste (MWAS) and tear-offs (TOAS). TOAS is much more available because it is the waste from re-roofing or roof removal projects, whereas MWAS is generated by the industry (Zhou, Button and Epps 2012). MWAS tends to be a preferred material due to its known composition, fewer contaminants and deleterious materials and lower stiffness. Zhou et al. (2013) studied several locations in Texas and found that the average high-temperature grade of the binder in TOAS was 175 °C, whereas that for MWAS binders was 131 °C.

Considering the stiffening effect of recycled materials in HMA and the related increase in the mixture cracking susceptibility, several strategies have been explored to address these issues in RAP/RAS mixtures. The main strategies include the following (Zhou et al., 2015):

- Limiting RAP and RAS use by percent allowed in mixtures
- Using soft virgin binders mainly with regard to low-temperature PG grade (PGL)
- Rejuvenating RAP and RAS binders with RAs

2.2. Effect of RAs on Binders with High RBRs

The option of rejuvenating RAP and RAS aged binders with RAs is now receiving increased attention by the academic community and the pavement industry due to associated technical benefits including, but not limited to, rheological restoration of the aged binder, improved workability of the mixture both in the laboratory and field, and ability to modify the PG grade of the binder blends. This subsection discusses the effects of RAs on binders with high RBRs.

The high- and low-temperature grades, PGH and PGL, respectively, of the blended binder are affected by the individual contributions from RAP and RAS binders as a result of the differences in stiffness and composition. In order to minimize their stiffening effect, the use of RA is crucial for reducing the PG grade of the blended binders (Shen and Ohne 2002; Shen, Amirkhanian, and Miller 2007; Tran et al. 2012; Mogawer et al. 2013; Zaumanis et al. 2014; Shen, Amirkhanian, and Lee 2005; Shen, Amirkhanian, and Tang (2007).

Some research has addressed the softening process of RA on the aged binders. Nevertheless, a deeper understanding of the working mechanisms is required. According to Tran et al. (2012), the working mechanism mainly depends on the uniform dispersion of the RA within the mixture and the diffusion of the RA into the aged binder. In particular, physical processes are the principal cause for the dispersion produced by mechanical mixing. The mixing time is an important factor related to the efficiency of the RA in the recycled mixture, and diffusion is the process where a constituent moves from a higher concentration to a lower concentration. For the diffusion mechanism, the

RA spreads into the aged binder in the following four steps (Carpenter and Wolosick 1980):

- The RA forms a very low viscosity layer that surrounds the aged binder that is coating the recycled material and aggregates.
- The RA begins to penetrate into the aged binder layer, softening the aged binder. The amount of the RA surrounding the recycled material particles decreases as penetration continues.
- Penetration of the RA into the aged binder continues, decreasing the viscosity of the inner layer and increasing the viscosity of the outer layer of the recycled material particle.
- Equilibrium is approached after a certain time.

The penetration of the virgin and aged binder (composing the blend) is influenced by the diffusion characteristics of the RA in the aged binder. Also, it has a significant effect on the performance of the resulting binder and mixture. From the compositional perspective of the blends, Karlsson and Isacsson (2003) pointed out that the rate of diffusion is governed by the viscosity of the maltene phase and not the viscosity of the entire aged binder.

Furthermore, the degree of aging, or stiffness, of the aged binders in the recycled materials is one of the most influential factors that control the effectiveness of the RA in restoring certain physical and chemical properties of the aged binders. The higher the stiffness of the aged binder, the higher the dosage and the lower the viscosity of the RA needed to restore the aged binder properties.

2.3. The Concept of Blending

This subsection discusses the definition of blending and its relationship to recycled asphalt blends. The degree of blending is defined as the percentage of aged binder (contained in the recycled materials, for instance RAP and RAS) that is effectively active within the mixture (i.e., contributes with the virgin binder to bond the aggregate). It depends upon the aged binder content, difference in stiffness between the aged and virgin binder, and the aggregate gradation in the recycled material (Coffey et al. 2013). In this respect, major research needs are focused on characterizing the degree of blending occurring in mixes with high RBR. This is relevant because the mixture volumetric properties and performance are tied to the degree of blending; therefore, inadequate assumptions in mixture design might lead to scenarios in which the pavement exhibits distress prematurely. For instance, assuming a higher degree of blending leads to lower virgin binder content that will produce a stiff mixture that is more prone to fatigue and thermal cracking. The opposite is also likely to occur, leading to a softer mixture with higher susceptibility to rutting (Coffey et al. 2013). Moreover, Copeland et al. (2010) state that low-temperature pavement cracking in recycled mixtures is mainly caused by a poor blending or lack of blending between the virgin and aged binders.

The current standard specification for Superpave volumetric mixture design, American Association of State Highway and Transportation Officials (AASHTO) M323, assumes that complete blending occurs between the virgin and aged binders during HMA production (i.e., forming a perfectly homogenous blend), and many state agencies assume this full blending condition. However, in most cases, the degree of blending is partial (Coffey et al. 2013; Kriz et al. 2014), and there is no standard method available to determine the exact degree of blending. Moreover, current methods employed in the laboratory mixture design process in order to include high RBRs in recycled mixtures may not be representative of plant operations due to the fact that the plants operate under conditions less controlled than those encountered in the laboratory, and this affects the blending between aged and virgin binder (O'Sullivan 2011). Kriz et al. (2014) explained four possible scenarios of virgin-aged binder contact (due to mechanical mixing) and blending (due to diffusion) in mixtures. Four possible scenarios are valid for the blending occurring among the virgin and recycled materials. In particular, good contact between all the components in the mixture is needed to guarantee that the rheological and mechanical properties are consistent to allow for appropriate performance in the field.

2.4. Aging Characterization

After introducing the concept of recycling, the use of RA in recycled mixtures, and the concept of blending, it is also relevant to recall that HMA is subjected to aging (i.e., increase in stiffness and reduction of elastic response as a result of volatization and chemical oxidation) in the field induced by the environmental conditions to which the pavement structure is subjected. In this regard, characterizing aging in the component materials of HMA constitutes an important element for better understanding the rate at which these changes occur and the effect that adding RAs can have on the aging evolution. This section provides a recently developed approach to characterize aging of binders that can also capture the effects of RAs.

The viscoelastic response of binders is typically captured at different loading frequencies and temperature conditions by means of two rheological parameters (i.e., stiffness and phase angle). In particular, the stiffness is measured as the shear complex modulus (G*) at high and intermediate temperatures and asphalt binder stiffness (S) at low temperatures. The phase angle is characterized as δ at high and intermediate temperatures and m-value at low temperatures. These parameters are affected by aging that causes age hardening or stiffening, producing increased stiffness and reduced phase angle. Recent research has led to the discovery that these two parameters can be used in the characterization of aging embrittlement by plotting their response at a particular temperature and frequency on a Black Space diagram (Glover et al. 2005; King et al. 2012). In this diagram, each point represents an aging state that evolves from the lower right to the upper left, indicating a progressive increase in G* and decrease of δ.

Black Space diagrams also depict a damage zone where cracking likely begins due to brittle rheological behavior defined by an intermediate-temperature DSR parameter called the Glover-Rowe (G-R) parameter that was originally defined by Glover et al. (2005) as the DSR function ($G'/(\eta'/G')$) and reformulated for greater practical use by Rowe (2011) in a discussion of Anderson et al. (2011) as $G'/(\eta'/G')/\ \omega = G^*\ (\cos\delta)2/(\sin\delta)$, where all rheological properties are referenced to 0.005 rad/s and 15 °C. The G-R values between 180 and 450 kPa correlate to low ductility values of 5 cm to 3 cm, respectively. These limits were previously related to surface raveling and cracking by Kandhal (1977).

3. EXPERIMENTAL DESIGN

In previous sections, an introduction to the use of recycled materials on HMA and a summary of the concepts of the effects of aging and RAs on binders with high RBR were provided, along with a recent approach to capture these effects. This section presents the experimental design, which includes a description of the field project, the testing plan, and the laboratory test description.

3.1. Texas Field Project

Texas State Highway 31 (SH 31) reconstruction project (Texas Department of Transportation [TxDOT] Project ID 06401068) included an approximately 1.4-mile asphalt overlay placement. This project had five test sections to study and evaluate the effects of different rejuvenators on the performance of recycled mixtures with high RAP and MWAS content. Originally, this project was initiated under a research study sponsored by TxDOT. The overlay was constructed in the first week of June 2014.

At this project site, SH 31 is a divided highway with two lanes in each direction. These test sections are located between the east side of the city of Murchison and the west side of the city of Brownsboro, Texas. All five sections have identical pavement structure: 1-inch crack attenuating mixture covered by 2-inch dense-grade Type C mixture. Each of the five test sections had a different surface mixture, as shown in Table 1.

Table 1. Test Sections with Five Different Mixtures.

Section No.	Mixture Type and Recycling Agent Type	Asphalt Binder Type	Description
1	Virgin Mixture	PG 70-22	Virgin aggregate
2	Control Mixture	PG 64-22	0.3 RBR 10% RAP and 5% MWAS
3	Recycled Mixture with T1		

3.2. Testing Plan

A summary of the materials and testing conditions is presented in Table 2. Laboratory tests included: (1) determination of high-temperature PG grade for determining RA dosage; and (2) master curves, G-R parameter, and FTIR at original, short-term aging (rolling thin film oven [RTFO] test) and three long-term aging conditions (pressure aging vessel [PAV] at 20, 40, and 80 h) for the rheological and aging experiments. Each of the tests in Table 2 was conducted on two replicates. As depicted in Table 2, additional RBRs to those presented in Table 1 were included in the laboratory experiments, which were intended to evaluate the effects of T1 on blends with RBRs exceeding 0.3 and compare their rheological and aging performance. In addition, an extra virgin binder, PG 64-28, was included in the experiment with the intent to include another binder source with a different low-end PG grade and to determine performance-based differences among the materials in Table 2. The corresponding test plan is presented in Figure 1.

Table 2. Binder Blend Experiment for Recycling Agent Type T1.

Virgin Binder	RBR (RAP|RAS)	RAPBR and Source	RASBR and Source	RA	PGH and PGL @ 0, 2 & 10% RA	Master Curves Glover-Rowe (G-R) parameter FTIR				
						Original	RTFO*	PAV** 20 h	PAV** 40 h	PAV** 80 h
64-22	0	-	-	Tall Oil T1	✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓
	0.3	0.1 TX	0.2 MWAS		✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓
	0.4	0.4 TX	0.0 MWAS		✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓
	0.5	0.25 TX	0.25 MWAS		✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓	✓✓✓
64-28	-	NH	-		-	✓	✓	✓	✓	✓
70-22	-	TX	-		-	✓	✓	✓	✓	✓

*RTFO: Rolling Thin Film Oven Test (ASTM D2872)

** PAV: Pressure Aging Vessel (ASTM D6521-13)

T1 is a tall oil product fabricated from natural raw materials that allows for reduction in the viscosity to meet binder specifications and reduced compaction temperatures in the field. The common properties of T1 are:

- Viscosity @ 140 °F (60 °C): (cPs) < 100 cPs (ASTM D3381 / D3381M-13)
- Flash Point Cleveland Open Cup: °F > 420 °F (ASTM D92-12b)
- Specific Gravity: 0.92 to 0.95 (ASTM D891-09)
- Water Content: wt% < 2.0 wt% (ASTM D2216-10)

- Appearance: Dark amber liquid at room temperature

3.3. Laboratory Tests Descriptions

The laboratory characterization of the materials presented in the previous subsection involved PG grading with DSR, FTIR, G-R parameter in Black Space diagrams, and master curves. Further details regarding these tests are subsequently provided. The test plan includes the major tasks presented in Figure 1 and explained in the following subsections.

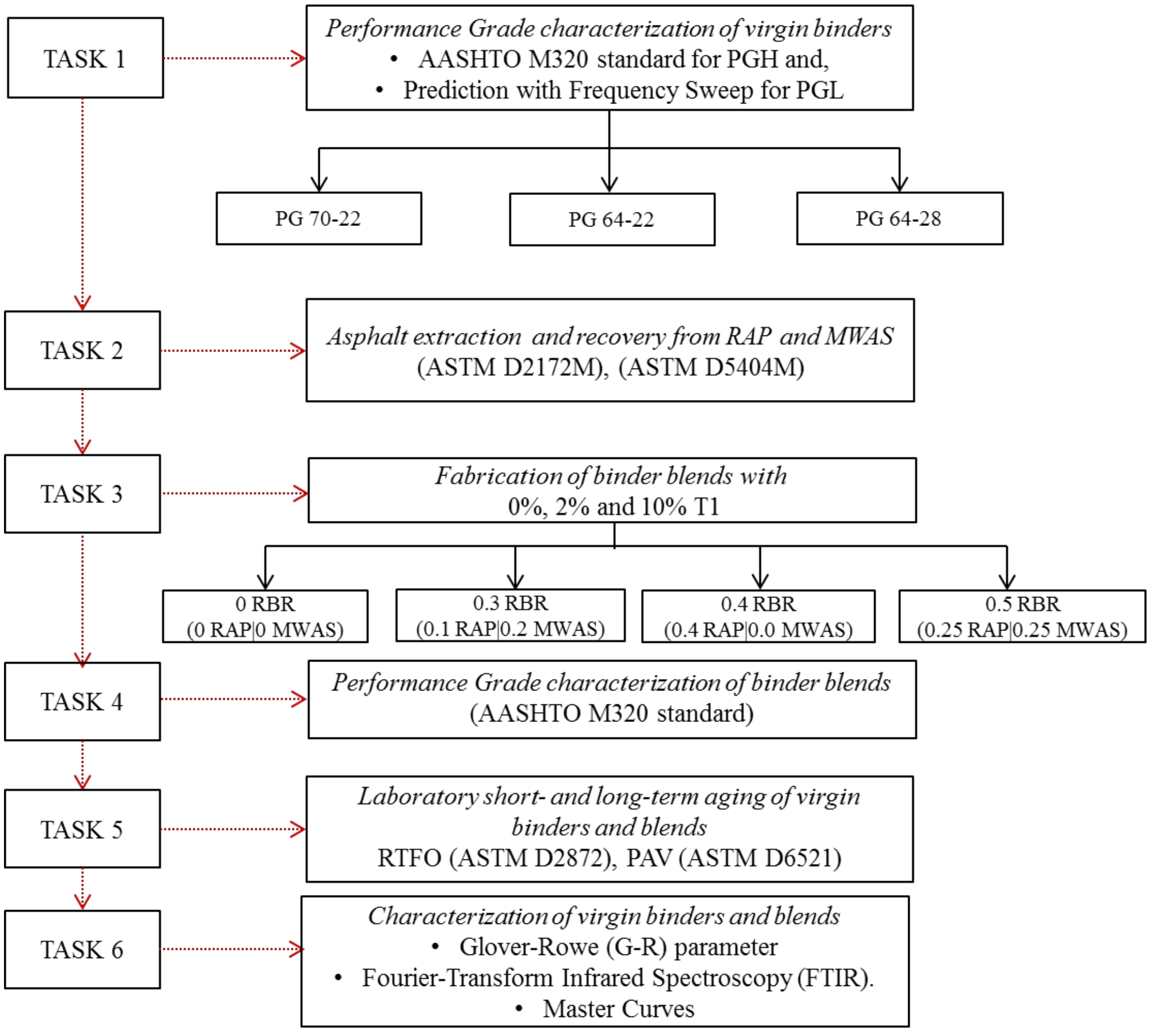


Figure 1. Test Plan Phases Involved in the Characterization of the Binder Blends.

3.3.1. PG grading with DSR

A Malvern research-grade DSR (Figure 2), Kinexus Pro model was used for the PG grading, G-R parameter, and frequency sweeps required for the master curves. Determination of PG grade followed the AASHTO M320 standard for PGH. PGL was not determined for recycled binders due to their extremely high stiffness and equipment limitations.

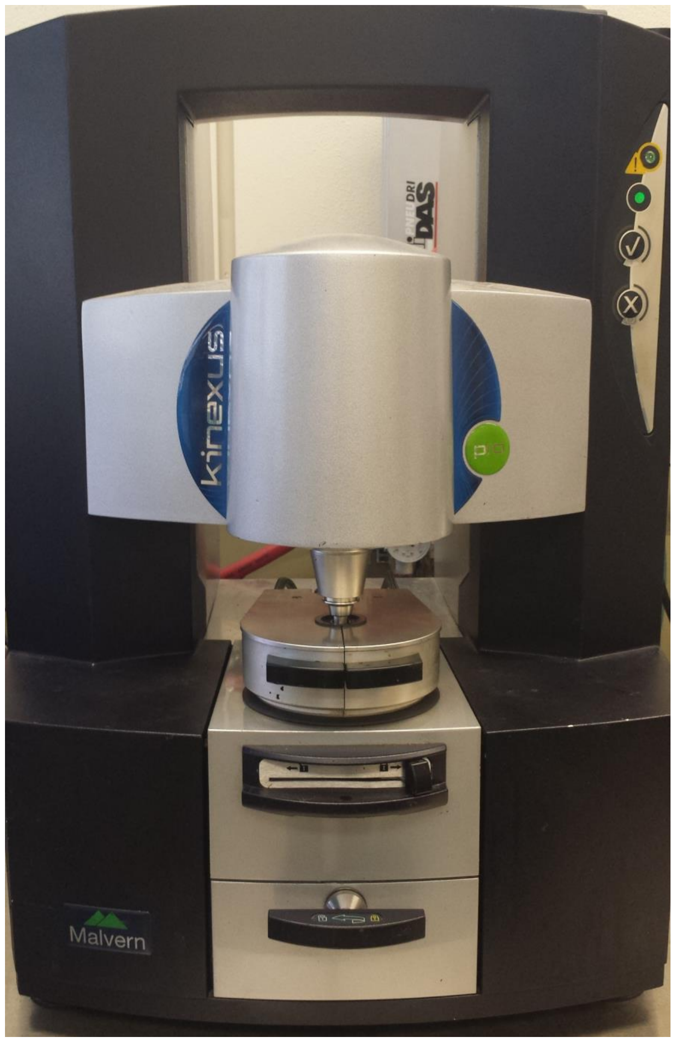


Figure 2. Dynamic Shear Rheometer.

3.3.2. Aging protocol

Due to the addition of RAP and MWAS in blends with high RBRs, stiff binder blends were obtained. For the virgin binders and binder blends included in this study, various artificial aging conditions were selected that included short- and long-term artificial aging. The former was conducted in RTFO (ASTM D2872) and the latter in PAV (ASTM D6521), including 20, 40 and 80 hours. These aging times were selected based on previous literature studying cracking in Black Space diagrams and G-R parameter for asphalt binder after 20 hours of PAV aging (King et al. 2012; Zhou et al. 2015; Mogawer et al. 2015; Mensching et al. 2015).

3.3.3. Fourier transform infrared spectroscopy

A Bruker Tensor 27 FTIR instrument was used for characterizing the aging occurring in the binder blends by determining the carbonyl area (CA) after short-term aging in RTFO and long-term aging in PAV (Figure 3). CA was determined as the area under the curve of a specific wavelength range in the absorbance spectrum (1820.50–1650.79 cm^{-1}) due to its correlation with oxidative aging characteristics in virgin binders and other materials used in the asphalt industry (Lamontagne et al. 2001; Glover et al. 2005; Zhou et al. 2015).

Figure 3. Fourier Transform Infrared Spectroscopy.

3.3.4. Glover-Rowe parameter in Black Space diagrams

The G-R parameter offers an indication of the cracking resistance of asphalt binders at intermediate temperature (Rowe 2011; King et al. 2012). This fatigue cracking parameter, $G'/(\eta'/G')$, was initially derived from the Maxwell model by Glover and correlated to binder ductility at intermediate temperature (15 °C, 1 cm/min) (Glover et al. 2005). In addition, Kandhal (1977) did previous field-testing to tie binder ductility to age-induced cracking. In 2011, Anderson et al. conducted laboratory testing and confirmed the correlation between Kandhal's ductility and Glover's fatigue parameter (Anderson et al. 2011). Theoretically, once a binder goes into or above the damage zone in the Black Space diagram (between the cracking onset curve at G-R = 180 kPa and the cracking damage zone at G-R = 450 kPa), its cracking resistance at intermediate temperature is compromised, meaning that the binder will likely exhibit fatigue cracking, starting with micro cracks that could later turn into macro cracks (alligator cracking). At this level of aging, the material is very brittle and the structural integrity of the pavement structure containing that binder type is at stake, not only due to its susceptibility to block cracking but also to surface raveling and, possibly, moisture damage (King et al. 2012).

With regard to Black Space diagrams with G-R, there is still little knowledge about the correlation of this cracking parameter with field performance, considering that several aging conditions are involved in the characterization and that those must have an equivalent of field aging. For instance, indicating that PAV-20 is equivalent to 7 to 10 years of aging in the field may be a good assumption based on previous research on the topic (Anderson et al. 1994; Bahia and Anderson 1995; Roberts et al. 1996); however, indicating the equivalent scale for the other simulated aging conditions in the laboratory to the field may be inaccurate and may not provide an indication as to what level of cracking might really affect the pavement structure.

3.3.5. Master curves

The DSR was used for characterizing the asphalt binders at high (60, 64, and 70 °C, from 0.001–100 rad/s), intermediate (22 to 46 °C in 6 °C increments, from 0.01–100 rad/s), and low (15, 10, 4 and −2 °C from 0.01–100 rad/s) temperatures. RHEA™ software from Abatech was used for analyzing the DSR data due to better fits than the manual methods obtained from Microsoft Excel® sheets. Subsequently, it was used to produce the master curves by using time–temperature superposition and determine some rheological properties discussed subsequently. The Williams–Landel–Ferry (WLF) model with an arbitrarily selected temperature of 25 °C was used in the master curves construction. Additional properties were calculated from master curve data, in addition to the shape and location of the master curves, in order to characterize the linear viscoelastic properties of the blends included in the study (Huang et al. 2014). These included the following: low-shear viscosity (LSV) $\left(|\eta^*| = \sqrt{(\eta')^2 + (\eta'')^2}\right)$, DSR function (DSRFn) $\left(\frac{G'}{\left(\eta'/_{G'}\right)}\right)$, Glover-Rowe parameter $\left(G - R = \frac{G^*\omega cos^2\delta}{sin\delta}\right)$, and crossover modulus and frequency (when the phase angle equals 45°, analogous to the rheological index, R). The LSV is defined as the complex viscosity at very low frequency, and is an approximation for the theoretical zero shear viscosity value (Naskar et al. 2013). LSV was estimated at 0.001 rad/s and 60 °C from master curve data in RHEA. The data were subsequently processed in Excel for comparisons and corresponding discussions.

4. TEST RESULTS AND DATA ANALYSIS

This section presents the main results from the laboratory tests conducted on binder blends fabricated with recycled asphalt pavement, manufactured waste asphalt shingles, and one tall oil recycling agent (T1). Subsequently, this section focuses on giving evidence of the effect of the recycling agent T1 on the rheology of binder blends with high RBRs. The section is composed of two main subsections: (1) virgin binders, and (2) binder blends that show PG grading determinations, dosage calculation to restore PGH and PGL, aging characterization, and master curve data.

4.1. Virgin Binders

4.1.1. High- and low-temperature PG grade

As depicted in Figure 4, the laboratory tests conducted on three true replicates indicated that the virgin binders met their PG grade. The continuous PG grades were PG 74.3-24.5 (PG 70-22), PG 68.2-24.5 (PG 64-22) and PG 66.9-29.6 (PG 64-28). Intermediate temperatures were also determined as 19.2, 19.2, and 20.4 °C, respectively. In addition to the virgin binders, extracted and recovered binder from RAP and MWAS was PG graded, resulting in a continuous grade for PGH equal to 106.6 and 137.4, respectively. The low-temperature grade could not be determined either by bending beam rheometer (BBR) or DSR due to the high stiffness of the materials and limitations in the equipment’s capabilities.

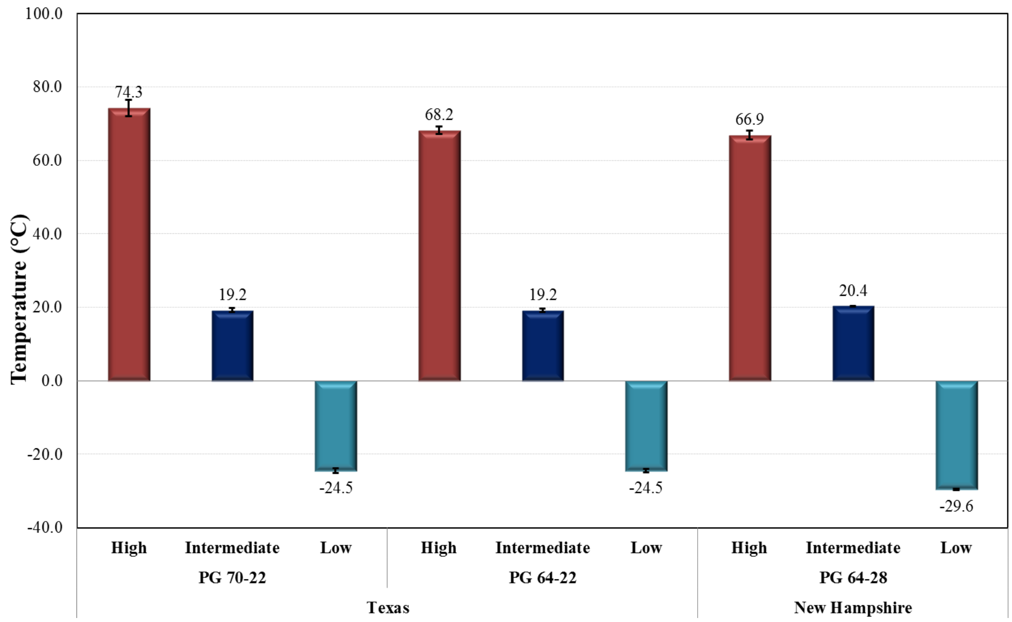


Figure 4. PG Grade Determination for the Three Asphalt Binder Types Included in the TX Cluster of Experiments.

4.1.2. Aging characterization

4.1.2.1. Softening effect

As depicted in Figure 5, the three binder types differ in aging behavior as characterized by the G-R parameter. The PG 64-28 binder exhibits much lower values for both phase angle (δ) and complex modulus (G^*) at the original and four aging conditions (i.e., RTFO, PAV-20h, PAV-40h, and PAV-80h). Conversely, the PG 70-22 binder clearly initiates in the Black Space diagram in a position where higher stiffness and lower phase angle are identified. In addition, Figure 5 depicts initial differences in the binder properties (G^*, δ) that are captured in the Black Space diagram. In fact, the binders initially have a high capacity to endure the tensile stresses due to their high tensile strength when unaged or little aging has occurred, and they locate on the lower right of the Black Space diagram (i.e., high phase angle and low stiffness). However, as aging progresses, the binders become more rigid and brittle and the tensile strains that the materials are able to endure are reduced as a result of lower tensile strengths, providing an explanation to the location (lines very close to each other for the three binders) in Black Space for the binders after 20 hours of PAV.

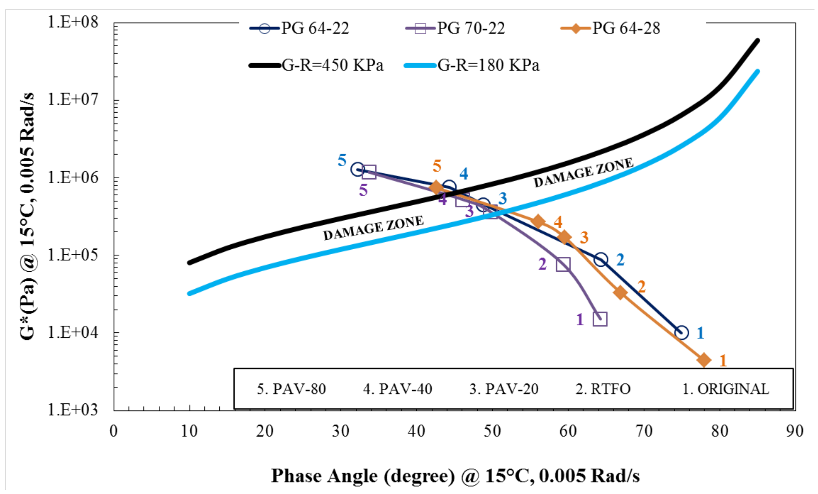


Figure 5. G-R Black Space Diagram for Virgin Unaged and Aged Binder.

As shown in Table 3, artificial aging in the PAV induced significant changes in the rheology of the virgin binders, as indicated by the percent increase from original condition (unaged material). A maximum of 16,735% increase of G* was reached after 80 hours of PAV aging for the PG 64-28 binder, whereas PG 70-22 had a 7,831% increase. Similarly, a decrease in the phase angle is expected for the materials after aging since the material gets stiffer and more elastic. The maximum decrease in phase angle was found for the PG 64-22, followed by PG 70-22 and PG 64-28 after PAV-80h aging. This information offers an indication of the predominant impact that artificial aging has on the rheological properties of virgin materials as evaluated by laboratory tests. Further field comparisons are required to establish correlations with laboratory data that lead to a deeper understanding of the equivalence of, for instance, 40 or 80 hours of PAV aging, to field aging occurring in the pavement structure.

Table 3. G-R Parameter Results for the Virgin Unaged and Aged Binders.

Binder Type	**Aging Condition**	***G* (MPa)***	***δ (°)***	**% Increase of G* from Unaged**	**% Decrease of δ from Unaged**	**% Increase of G* between Aging Condition**	**% Decrease of δ between Aging Condition**
PG 70-22	Original	1.51E+01	64.3				
	RTFO	7.69E+01	59.3	509	8	509	8
	PAV-20h	3.66E+02	49.7	2,419	23	476	16
	PAV-40h	5.25E+02	46.1	3,469	28	143	7
	PAV-80h	1.18E+03	33.7	7,831	48	226	27
PG 64-28	Original	4.44E+00	77.9				
	RTFO	3.29E+01	67	742	14	742	14
	PAV-20h	1.70E+02	59.6	3,821	23	515	11
	PAV-40h	2.69E+02	56.1	6,070	28	159	6
	PAV-80h	7.42E+02	42.6	16,735	45	276	24
PG 64-22	Original	1.01E+01	74.9				
	RTFO	8.81E+01	64.3	877	14	877	14
	PAV-20h	4.51E+02	48.8	4,489	35	512	24
	PAV-40h	7.54E+02	44.3	7,506	41	167	9
	PAV-80h	1.28E+03	32.2	12,765	57	170	27

Moreover, the information in Table 3 is presented graphically on a Black Space diagram to assess the cracking susceptibility of the materials at intermediate temperatures for very stiff materials (Figure 5). The label (1) corresponds to original (unaged material), (2) to RTFO and, (3), (4), and (5) to 20, 40, and 80 hours of PAV aging, respectively. As depicted in Figure 5 and summarized in Figure 6 after data interpolation, PG 64-28 was the binder that required more hours of artificial aging to reach both onset and significant cracking curves, followed by PG 70-22 with significantly lower values. Conversely, PG 64-22 required less time to reach both damage onset and significant cracking curves. As a result, PG 64-28 would be expected to perform better than the other two binders at intermediate temperature in terms of fatigue cracking. However, it does not imply that the other materials would not perform well due to the lack of correlation with mixture field performance and additional laboratory evaluations for fatigue resistance, as well as low-temperature cracking characterizations.

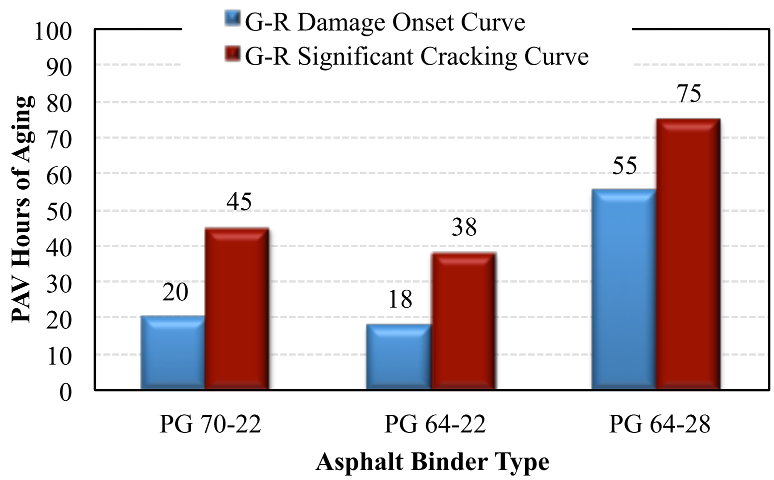


Figure 6. Comparison of Number of Aging Hours to Reach the G-R Damage Curves for Three Virgin Binders.

The G-R parameter in Table 3 presents the approximate number of PAV aging hours required for the three virgin binder types to reach the G-R damage onset $\left(\frac{G^*(cos\delta)^2}{sin\delta} = 180\ kPa\right)$ and significant cracking curves $\left(\frac{G^*(cos\delta)^2}{sin\delta} = 450\ kPa\right)$. The results indicate that PG 64-28 required more PAV aging hours to reach both G-R curves, thus this binder may have better cracking performance as compared to the other two binders at intermediate temperatures. Conversely, PG 64-22 had the lowest times to reach the curves in the Black Space, which may provide an indication of poor cracking performance as compared to the other two binders. Nevertheless, additional tests at low temperature, as well as comparisons to field performance are required to determine the *true* cracking performance of these binders. Furthermore, as shown in Figure 5, the 80 hours of PAV-aging for all three binder types produced very stiff materials with G* and δ values surpassing both onset and significant cracking curves, suggesting that this artificial aging condition abruptly changes the material rheological properties and makes it more prone to the appearance of fatigue cracking and, possibly, low-temperature cracking.

4.1.2.2. Aging resistance via FTIR carbonyl area

As depicted in Figure 7, the carbonyl area rate of increase was similar for the three virgin binder types, and a macroscopic comparison would suggest that the CA increases similarly among them (1.20% for PG 64-22, 1.11% for PG 70-22, and 1.01% for PG 64-28) or, in other terms, that binders produce CA at the same rate. Nevertheless, after statistical analysis, significant differences were found at the 95% confidence level, indicating that these had different rates of increase in CA as artificial aging time increases. Thus, adding to the previous analysis in Black Space for the virgin binders, a better aging and cracking resistance would be expected for the PG 64-28 as compared to the other two virgin binders used in the field project. However, it is important to note that the binders were produced from different sources and, therefore, their physicochemical composition makes them respond differently when subjected to laboratory testing and, thus, likely to perform differently in the field.

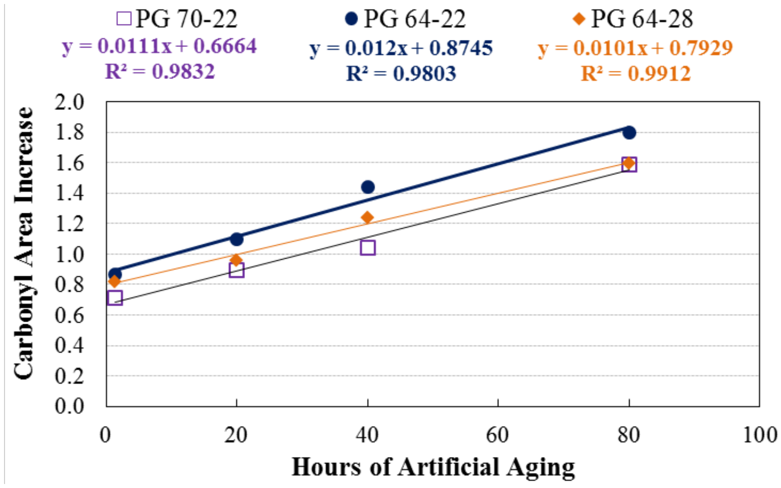


Figure 7. Comparison of Carbonyl Area for Three Virgin Binders.

4.2. Binder Blends

This subsection presents the experimental results for the PG grade characterization, dosage determination, and aging assessment through G-R parameter and FTIR for the asphalt blends. The objective was to evaluate the effect of RA dosage on the softening properties of the aged materials as measured by PGH and PGL, and to restore rheological properties in terms of stiffness and phase angle, as well as determine the effect of artificial aging on these results.

4.2.1. High-temperature PG grade

The linear regional-blend concept (Zhou et al. 2013) for multiple blends is studied in this subsection to determine the T1 dosage to restore the PGH. The dosages presented in Figure 8 include 0%, 2%, and 10%. Determinations of PG grade for the PGH followed the Superpave PG system. As depicted in Figure 8, and based on linear regression R^2 values ($R^2 = 0.999$ in all cases), a linear trend of reduced PG grade was found for increased recycling agent dosage, indicating the ability of T1 to soften the aged binders contained in RAP/MWAS. Furthermore, as observed, increased RBR requires increased dosages of RA as expected for stiffer materials.

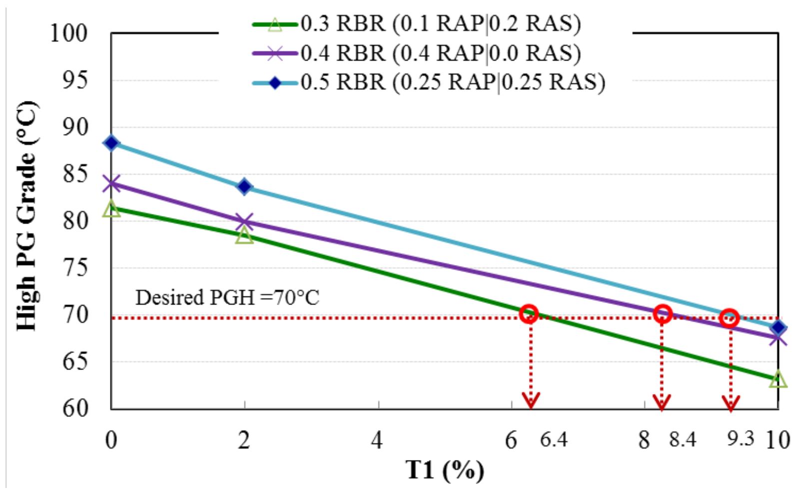


Figure 8. Comparison of High-Temperature PG Grade for Texas Binder Blends with T1.

4.2.2. Dosage determination

By using Figure 8, the dosage to restore PGH was determined after validating the linearity of the trends for the high temperature. The corresponding results are presented in Figure 9. As depicted, a minimum dosage was found for all the RBRs to restore the PGH. Then, the dosages are: 6.36 (0.3 RBR); 8.45 (0.4 RBR); and 9.28 (0.5 RBR). In addition, as observed in Figure 9, a trend of increased dosage is observed as RBR increases, which can be explained by the higher stiffness of the blend induced by the recycled materials (i.e., RAP and MWAS) that requires more recycling agent to reach a certain PG target in the high end of the spectrum.

In the case of high RBR mixtures, it would be expected that determination of PGL by BBR measurements is carried out in order to determine optimum dosages. However, there is still discussion in academic settings regarding the use of either PGH or PGL to determine optimum dosage due to the fact that different dosages are obtained, being lower for the PGH as compared to PGL. Thus, by considering that the use of RAs increases the overall cost of HMA production, using a lower dosage is evidently the most cost-efficient option. However, from the technical perspective, using higher dosages seems more appropriate for improved long-term performance of the HMA mixtures through: restoring binder rheology, improving the cracking resistance, and aging susceptibility. Nonetheless, caution has to be exerted to avoid causing rutting or moisture problems that can compromise the overall performance of the HMA over time.

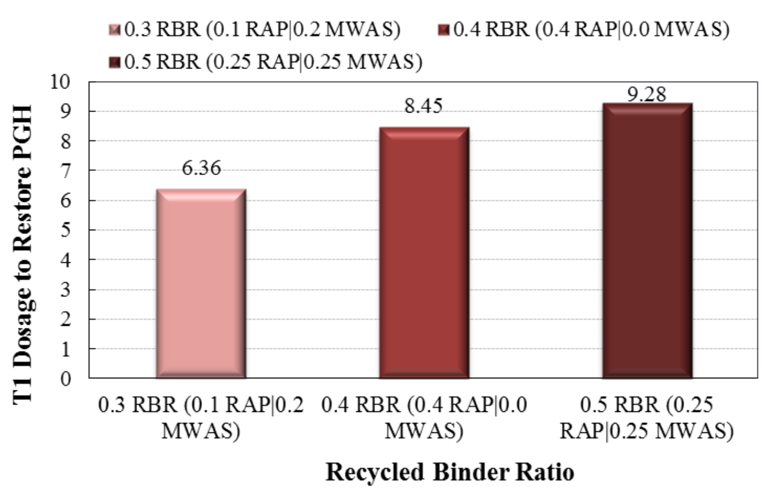


Figure 9. T1 Dosages to Restore PGH.

In section 5, a proposed methodology to include both PGH and PGL as tools for determining optimum dosages in binder blends at high RBRs and addition of RAs is presented. In that section, considerations on the importance of these tools and additional laboratory tests are included in an attempt to better characterize these materials in an efficient and reasonable manner.

4.3. Assessment of the Effect of RAP/MWAS/TOAS on Blends' High-Temperature PG Grade

This subsection focuses on the comparison of the effects of adding RAP, MWAS, and TOAS on the high-temperature PG grade for blends at different RBRs. The initial comparison involves the prediction of PGH for subsequent comparison with the measured PG grade following the AASHTO M320 standard. The comparison is developed considering the following conditions for the binder blends: (1) single addition of RAP, (2) single addition of MWAS to the blends, and (3) single addition of TOAS to the blends. The second comparison involves equal contribution from RAP/MWAS or RAP/TOAS. Finally, the third condition will evaluate the unequal contribution at 0.3, 0.4, and 0.5 RBRs for the aged materials in combination with the virgin binder and RAs.

4.3.1. Unbalanced addition of aged binders

As presented in Figure 10, predicted and measured values of PGH correlated reasonably well (R^2 = 0.97) with an average percent difference equal to 3.3%, indicating that the contribution of the individual components to the stiffness can be determined by simple mathematical calculations involving individual PGH, subsequently allowing for the analysis presented in the following subsections. More specifically, the prediction of PGH involved a weighted average of the individual contribution of the blends' components, using the measured PGH for virgin binders (PG 64-22, 69.4 °C and PG 70-22, 76.8 °C), RAP (108 °C), MWAS (133 °C) and TOAS (178 °C). For instance, for the 0.4 RBR blend composed by 40% RAP, 0% RAS, and the remaining 60% of virgin PG 64-22, the PGH calculation would be as follows:

$$Pred\_PGH_{Blend} = PGH_{virgin} * Percent_{virgin} + PGH_{RAP} * Percent_{RAP} + PGH_{RAS} * Percent_{RAS}$$

$$Pred\_PGH_{0.3\ RBR\_Blend} = 69.4 * 0.7 + 108 * 0.1 + 133 * 0.2 = 85.98\ ^{\circ}\ C$$

$$Pred\_PGH_{0.4\ RBR\_Blend} = 69.4 * 0.6 + 108 * 0.4 + 0 = 84.84\ ^{\circ}\ C$$

$$Pred\_PGH_{0.5\ RBR_{Blend}} = 69.4 * 0.5 + 108 * 0.25 + 133 * 0.25 = 94.95\ ^{\circ}\ C$$

A similar procedure was conducted for the other conditions shown in the following subsections. Therefore, comparisons of PGH for blends with the single addition of RAP, MWAS, and TOAS are conducted with the objective of better distinguishing the contribution of aged binders to the overall stiffness of binder blends at high RBRs.

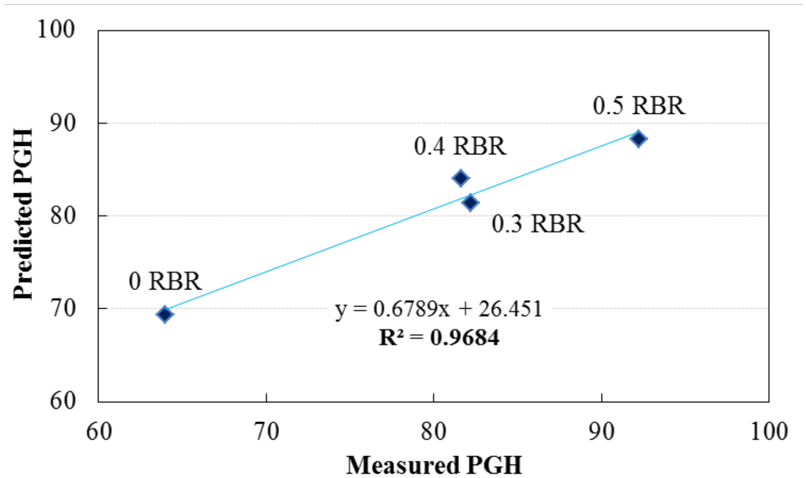


Figure 10. Comparison of Predicted and Measured PGH for the TX Asphalt Blends.

4.3.2. Single addition of aged binders

Taking into consideration the previous analysis for predicted versus measured PGH, this subsection focuses on evaluating the individual contribution of aged materials on the PGH for the blends at RBRs varying from 0 to 100. Through this analysis and as depicted in Figure 11, it can be observed that TOAS contributes significantly to higher stiffness in the binder blends, as would be expected for a very stiff material that has been subjected to multiple aging during production and to environmental conditions (i.e., oxygen exposure, moisture, and sunlight) when in service on roofs. Similarly, MWAS has a significant impact on the blends' stiffness as compared to RAP, mainly due to their differences in initial stiffness, conditioned by production aging and life cycle exposure to the environment. Furthermore, as presented in Figure 11, the differences in the individual contribution start increasing and being more evident at 0.3 RBR, which suggests that caution has to be exercised when using these materials for the fabrication of recycled mixtures at RBRs above 30%. The differences between the Texas virgin binders included in this study are barely noticeable as depicted in Figure 11, especially at RBRs above 50%. The predominant contribution of the MWAS and TOAS on the binder blends can also play a crucial role on the efficiency of rejuvenation when adding RAs, given that their effect might be reduced by the very stiff and scarcely comingled materials into the blend.

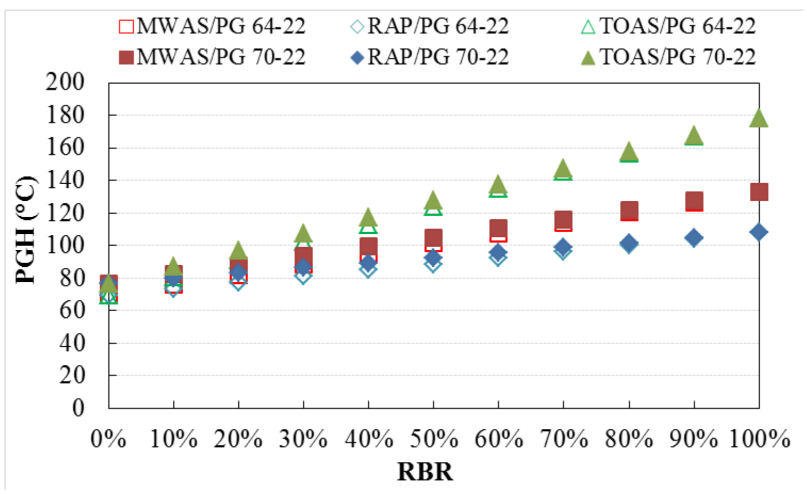


Figure 11. Comparison of the Single Addition of Aged Binders to the PGH with Virgin Binders PG 64-22 and PG 70-22 at Different RBRs.

4.3.3. Balanced addition of aged binders

As previously discussed, addition of a single aged material to binder blends at different RBRs has a significant impact on the binder blends' stiffness for both blends manufactured with PG 64-22 and PG 70-22 virgin binders. However, although addition of a single aged material is common in some locations, use of both RAP and RAS has gained attention recently. Therefore, this subsection attempts to discretize the contribution of the balanced addition by binder replacement of the combinations RAP-MWAS and RAP-TOAS.

More specifically, the contribution of TOAS under conditions of balanced addition of aged materials still induces higher stiffness in the blends, providing indication of the predominant role of TOAS as a stiffener of the blend and possibly suggesting that rejuvenation through the addition of RAs would be achieved only at higher dosages, thus, implying higher costs for manufacturing the asphalt mixes and also compromising performance due to the difficulties associated with the blending of TOAS with virgin and softer materials.

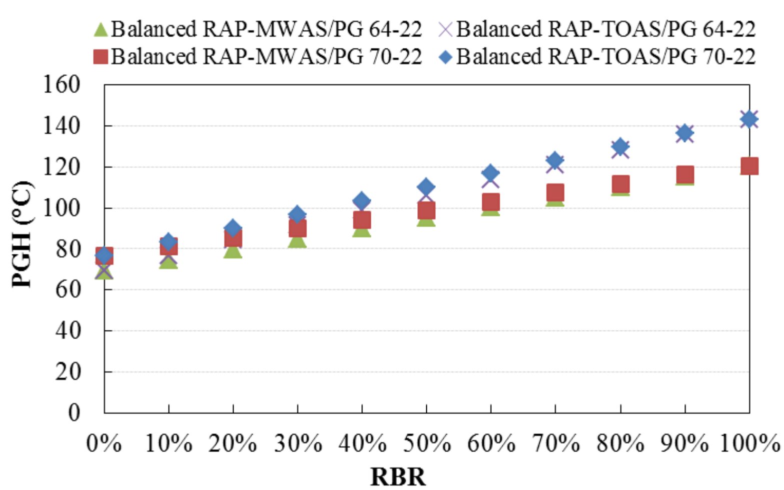


Figure 12. Comparison of the Balanced Addition of Aged Binders to the PGH with Virgin Binders PG 64-22 and PG 70-22 at Different RBRs.

4.4. Aging Characterization

This subsection presents the aging characterization results for the 0 RBR, 0.3 RBR, 0.4 RBR, and 0.5 RBR blends, fabricated with MWAS, RAP, and recycling agent, T1. It includes the softening via G-R parameter in Black Space, rheological restoration as evaluated with master curve data, and aging resistance via FTIR carbonyl area measurements.

4.4.1. Softening effect

Figure 13 shows the G-R parameter in Black Space, depicting a clear softening effect of T1 on the blends that increases in dependence to the T1 dosage. As observed, in all RBRs the 10% T1 blends have locations in Black Space to the lower right as compared to the 0% and 2% T1. In addition, the Black Space diagram provides some indication on the cracking susceptibility of the blends that depends on the position above or below the damage zone (G-R damage onset $\left(\frac{G^*(cos\delta)^2}{sin\delta} = 180\ kPa\right)$ and significant cracking curves $\left(\frac{G^*(cos\delta)^2}{sin\delta} = 450\ kPa\right)$). In this regard, more than 40 hours of PAV aging show a high cracking susceptibility for the materials shown. As depicted, when artificial aging reaches 80 hours, the materials have gone through the damage zone, indicating that these very stiff materials (high complex modulus and low phase angle) are more prone to cracking onset and propagation than those with 10% T1 addition. Moreover, the 0.4 RBR and 0.5 RBR blends started in Black Space above the damage zone even before PAV aging, offering a clear indication of extremely stiff materials at intermediate temperature and low frequencies.

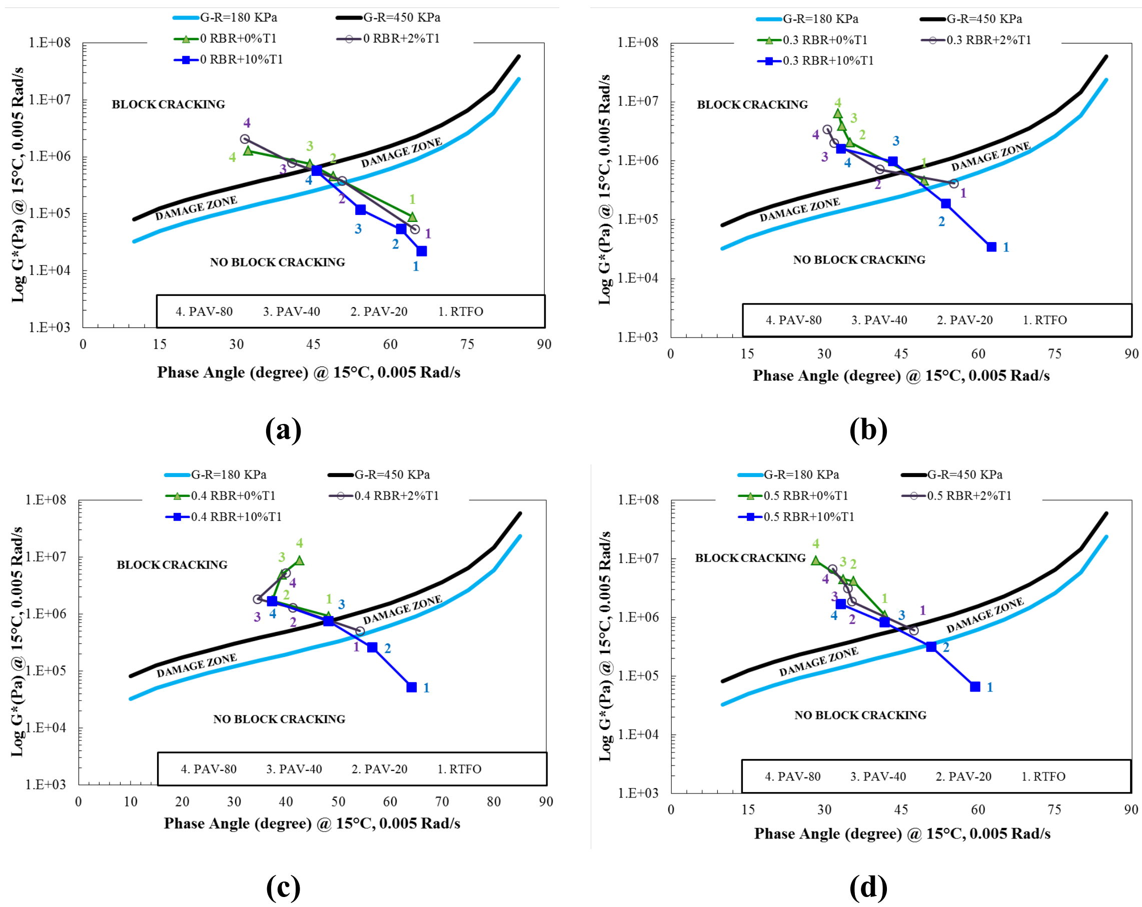


Figure 13. Black Space Diagram for Texas Blend with (a) 0 RBR (0.0 RAP| 0.0 RAS), (b) 0.3 RBR (0.1 RAP| 0.2 RAS), (c) 0.4 RBR (0.4 RAP| 0.0 RAS), and (d) 0.5 RBR (0.25 RAP| 0.25 RAS) and three T1 dosages.

Considering the cracking susceptibility of high RBR mixtures in RBR after artificial aging, caution needs to be exercised in selecting appropriate RA dosages that effectively restore the rheology and counteract the effect of aging on the aged materials and allow for improved cracking resistance. For instance, for all RBRs studied, the 10% T1 in Black Space clearly shows a considerable "distance" in terms of phase angle and complex modulus to approach the damage zone. Nevertheless, an optimum dosage needs to be used for characterizing G-R in Black Space in order to determine the approximate cracking susceptibility for specific conditions, since 10% could be far above the optimum and may influence other performance issues and also have a significant impact on the cost, as discussed previously.

4.4.2. Rheological restoration

This subsection presents master curve analysis to compare the effects of RBR, T1 dosage, and aging on the rheology of the blends. Only the full set is presented for the 0.3 RBR blend (0.1 RAP|0.2 MWAS). Figure 14 and Figure 15 show the comparison of master curves for each of the aging states presented in Table 1 and the three dosages studied for each blend (i.e., 0%, 2%, and 10% T1).

As observed in Figure 14 and Figure 15, the rheology of the blends is considerably affected by the short-term and long-term aging condition, as would be expected for highly aged materials with high stiffness values (G*) and low phase angles (δ). Similarly, the softening effect of T1 dosage on the rheology is appreciated in Figure 16 and Figure 17, consistent with the purpose of adding RA to blends with high RBR. The results also indicate that at higher frequencies the stiffness of the materials has similar values, whereas at low frequencies (i.e., long loading times), the differences among stiffness are much more evident. This provides information on the temperature influence on the mechanical response of the blends, indicating that at high temperatures and low frequencies the materials have evident differences in performance, while for the lower temperatures the materials respond similarly in terms of stiffness. Therefore, the master curve data clearly captured the influence of the T1 dosage and aging condition on the evolution of the complex modulus and phase angle in the given range of frequencies for the materials studied. More specifically, differences in performance are more likely to occur at high temperatures and low frequencies, as compared to low temperatures and high frequencies.

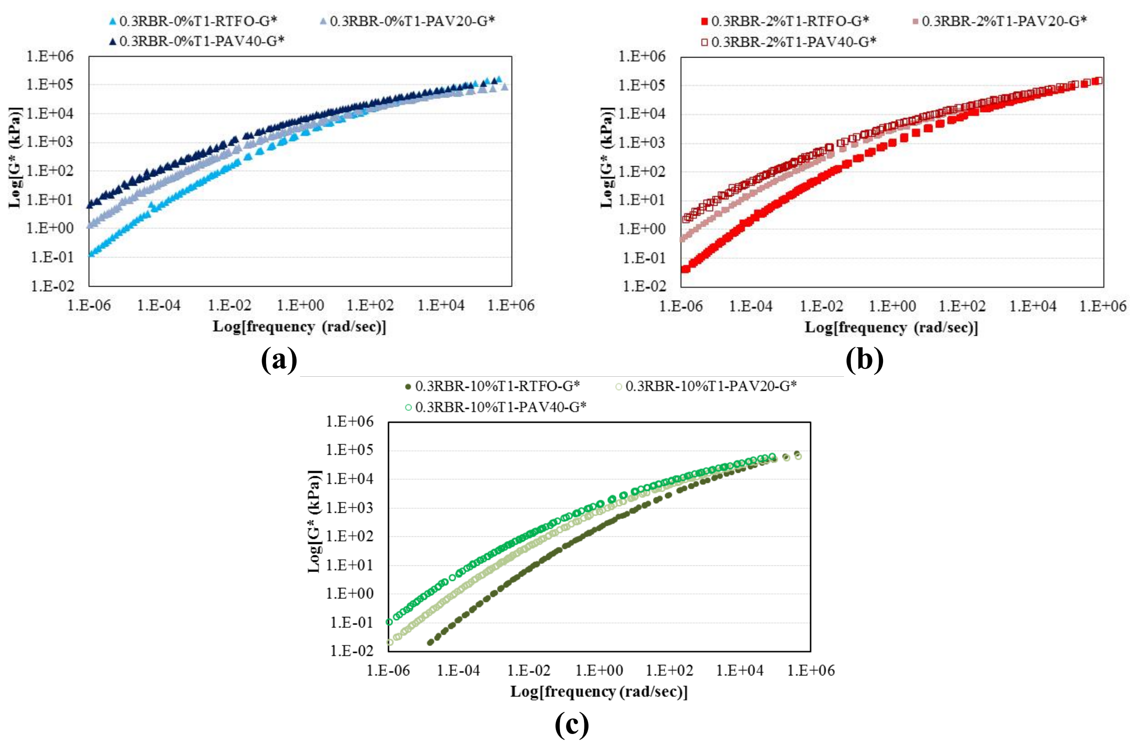


Figure 14. Complex Modulus Master Curves for the 0.3 RBR Blends at (a) 0% T1, (b) 2% T1, and (c) 10% T1.

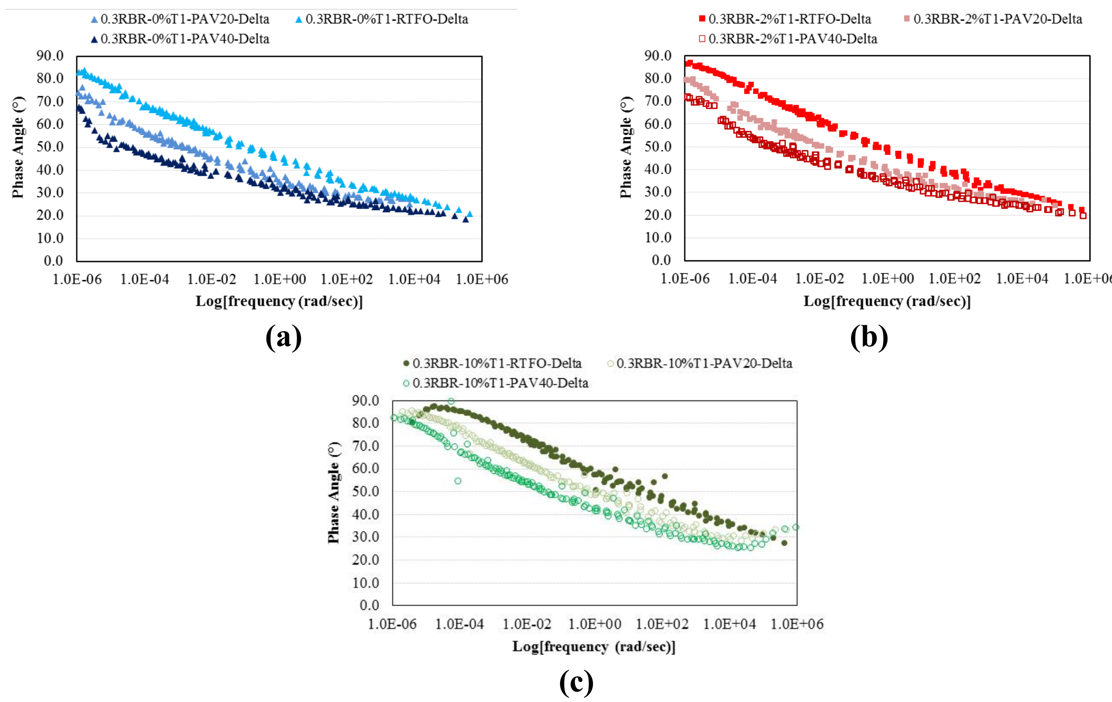


Figure 15. Phase Angle Master Curves for the 0.3 RBR Blends at (a) 0% T1, (b) 2% T1, and (c) 10% T1.

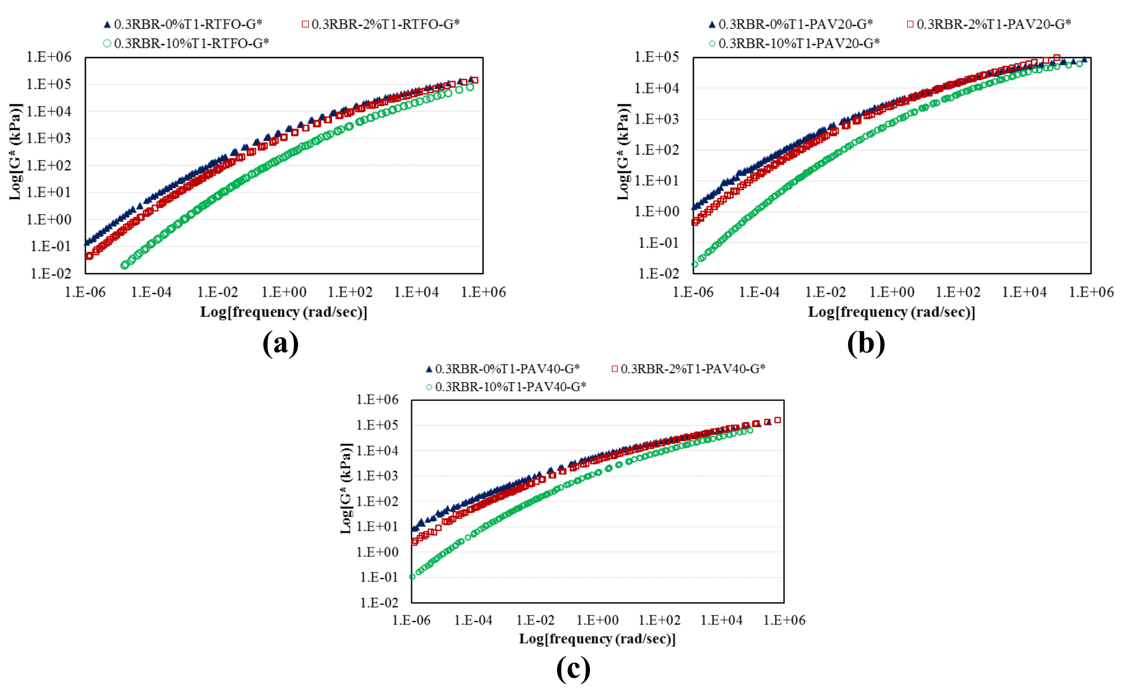
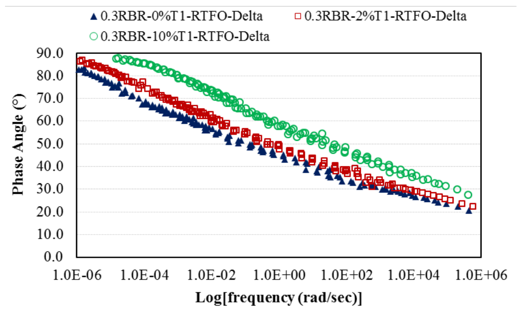
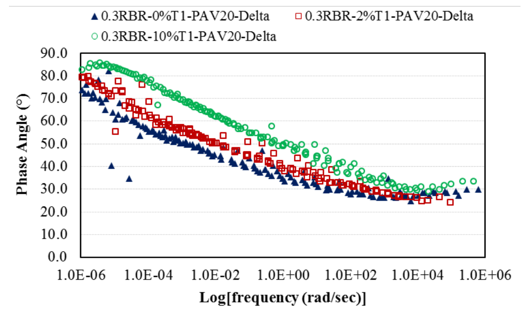


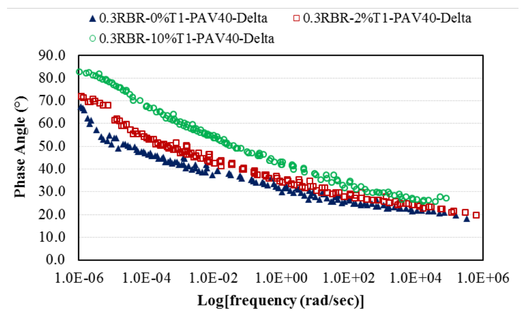
Figure 16. Complex Modulus Master Curves for the 0.3 RBR Blends at (a) RTFO, (b) PAV 20, and (c) PAV 40.



(a)



(b)



(c)

Figure 17. Phase Angle Master Curves for the 0.3 RBR Blends at (a) RTFO, (b) PAV 20, and (c) PAV 40.

Furthermore, by assessing the results presented in Figure 18, a clear effect of the dosage and aging condition on the rheological properties is observed, as indicated by LSV, DSRFn, crossover modulus, crossover frequencies, and G-R parameter. In particular, aging increases both LSV and DSRFn values, whereas dosage has an opposite effect, reducing their value. With regard to crossover modulus and frequencies (i.e., those that correspond to a phase angle equal to 45 degrees), aging increases these parameters, whereas the addition of RA induced the opposite trend. A similar trend is appreciated for the DSR function, which correlates to ductility at low temperature and also to CA growth, as presented in previous research by Glover et al. (2005). Conversely, an opposite trend for the effect of aging was found when evaluating the crossover modulus and frequencies. As previously evaluated by Partal et al. (1999), these parameters are important for characterizing the aging impact on the rheology and the compliance of the responses to the linear viscoelastic range, essential to make sure that the materials are being tested under the viscoelastic range, and no deformation or damage is being applied to these. The trends previously presented provide insights on the paramount role played by aging and dosage on the rheological properties of the 0.3 RBR blend with T1, as well as provide indication of the advantageous use of master curves to characterize G-R in the Black Space diagrams, as a strategy to save the amount of these materials. Previous research by Partal et al. 1999 indicated similar trends for these rheological properties as a function of aging.

A recent paper by Morian, Zhu, and Hajj (2015) presented a rheological evaluation of asphalt binders subjected to oxidation. Their approach involved LSV, DSR function or Glover-Rowe parameter, and dynamic shear crossover modulus. With respect to LSV, the authors evaluated an unmodified PG 64-22 virgin binder and indicated that there was a reduced susceptibility to aging by considering the slope after oxidation with respect to an unmodified PG 64-28. In addition, the authors emphasized the need to develop and use robust rheological indicators to characterize asphalt binders subjected to artificial oxidation in the laboratory, while simultaneously devoting more research efforts to better understand their correlation to particular distress types affecting pavement structures. In this regard, the use of master curves as a valuable tool for characterizing these materials was encouraged in order to obtain sound rheological data that can be used for a more comprehensive analysis of material responses.

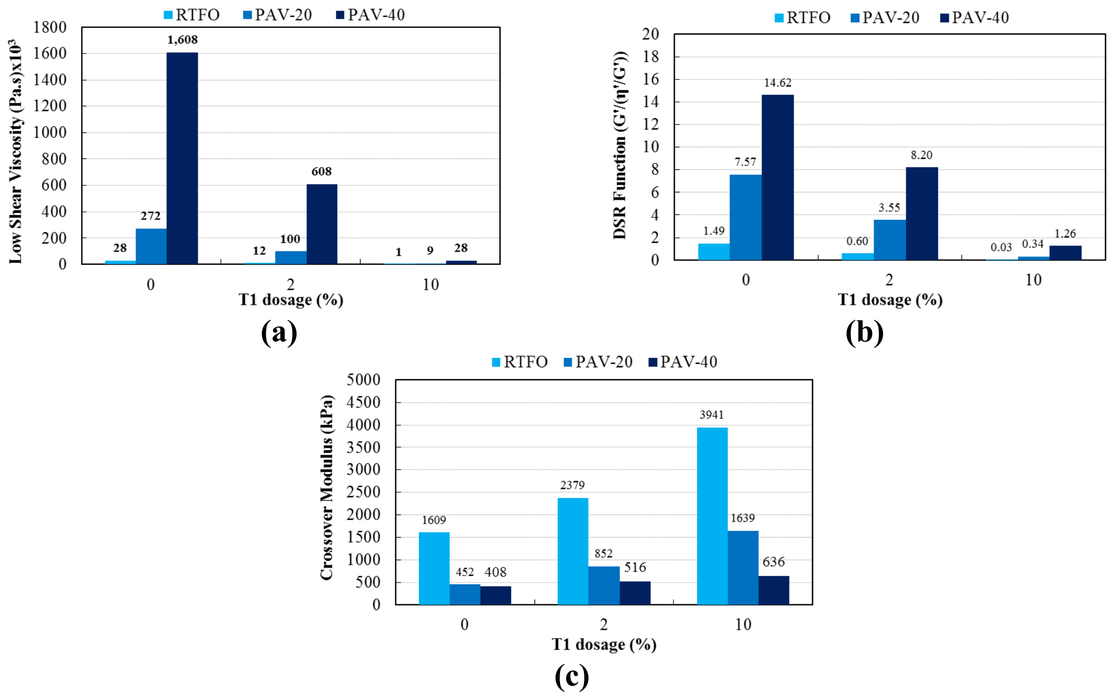


Figure 18. Rheological Properties for the 0.3 RBR Blend, including (a) LSV, (b) DSRFn, and (c) Crossover Modulus.

With respect to G-R parameter, the prediction via master curve data correlated well with experimental data obtained at 0.005 rad/s and 15 °C. As appreciated, R^2 values are approximately equal to 0.95 for both complex modulus and phase angle, supporting the validity of the data presented in Figure 19.

The master curves allow for a clear distinction with respect to the softening effect of the T1 dosage. As the dosage increases, the master curves' changes indicate a reduction in the complex modulus (G*) along the frequency range. Furthermore, the storage (G') and loss moduli (G'') indicate that both of them hold the time temperature superposition principle and can then be considered as thermorheological simple materials in the temperature range studied (−2 °C to 70 °C). These results are consistent with the purpose of applying RA to aged asphalt binders to soften it and restore its rheological properties (Partal et al. 1999; Rubab et al. 2011; Seidel and Haddock 2014; Xu et al. 2015).

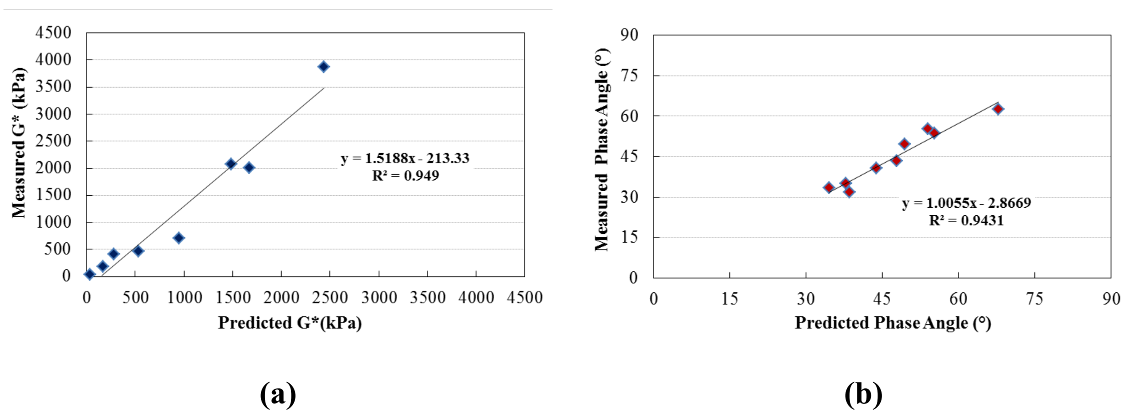


Figure 19. (a) Measured vs. Predicted G*, and (b) Measured vs. Predicted Phase Angle for the 0.3 RBR Blends.

As a result of the previous analyses, a clear effect of dosage and aging on the rheology was observed when a single RBR condition was evaluated, as well as when different RBRs were compared. In addition, a possible lack of softening effect of T1 on the rheology was identified after 20 hours of PAV aging, which might suggest that this RA is not efficient in the long-term for use in mixtures with RAP and MWAS. In section 5, a set of recommended tools and a methodology are provided based on the results from this and previous sections of the thesis.

4.4.3. Aging resistance via FTIR carbonyl area

As previously shown, short- and long-term artificially induced aging (RTFO and PAV, respectively) provoke rheological changes in the blends that depend on the dosage and the RBR. Nevertheless, aging evolution needs to be chemically characterized via laboratory testing. In this context, this subsection presents the blends characterization by means of FTIR CA. As observed in Figure 20, T1 dosage influenced the CA rate of increase over time of artificial aging. Similarly, a quantitative analysis of slope was conducted to determine the rate of increase in the CA. It indicated that the 2% T1 had the lowest average rate of increase in CA, as compared to the highest rate found for 10% T1.

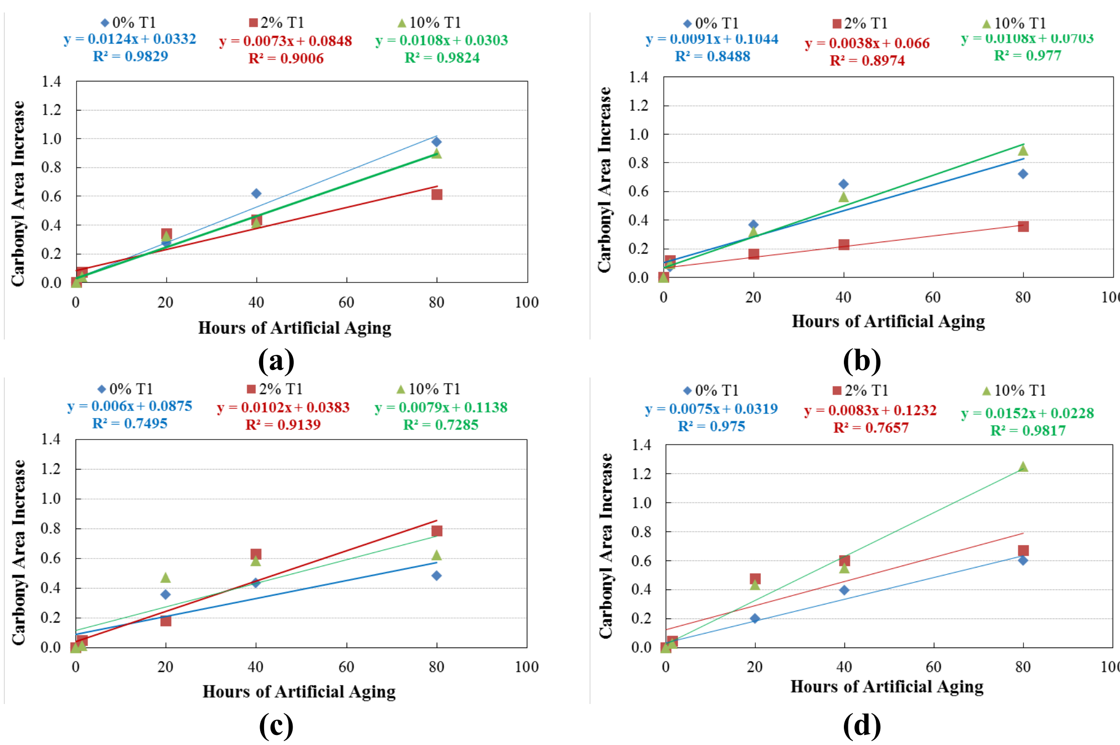


Figure 20. Comparison of Carbonyl Area for the (a) 0 RBR, (b) 0.3 RBR, (c) 0.4 RBR, and (d) 0.5 RBR in Original, RTFO, and PAV Aging Conditions.

Furthermore, as it would be expected for higher aging time, the CA increased in all cases. In addition, the relatively high rate of increase in CA for the 10% T1 could be explained by high CA values that were determined for T1 (7.23 a.u.) as compared to other RAs with values below 2.0 a.u. (Figure 21). Furthermore, from the chemical perspective, the composition of T1 includes high content of fatty acids, in which CA is high and occurs naturally in the original material after production; whereas the other RA evaluated have different chemical composition that includes paraffinic type and other categories of recycling agents. In particular, as indicated in previous research, fatty acids may include several chemical types, such as palmitic, stearic, oleic, linoleic, or linolenic, which depends on the source and type of process that the material underwent in the production plant (Seidel and Haddock 2014).

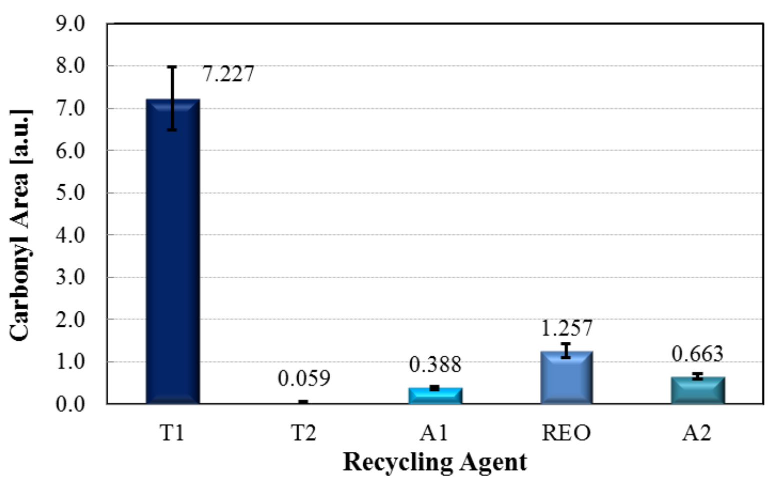


Figure 21. Comparison of Carbonyl Area for Different RAs.

5. CONTRIBUTION TO RESEARCH

This thesis presented a laboratory characterization of binder blends with high RBRs that included the aging and rheological assessment, conducted through various laboratory tests presented in the previous sections. The objective of this section is to propose a method for selection of optimum dosage that involves economical, technical, and environmental criteria.

Therefore, this section is comprised of two main subsections. The first will present a set of recommended tools for characterizing binder blends in an efficient and rational manner. The second will present the optimum dosage selection method and will include an example for the materials studied in this thesis.

5.1. Recommended Laboratory Tools to Characterize Binder Blends with High RBR

The information presented in Table 4 covers the main advantages and disadvantages associated with the testing conducted in the experimental design included in this thesis. Based on these, a recommended set of tools is provided for use in similar research on binder and binder blends with high RBRs.

Table 4. Advantages and Disadvantages Associated with the Set of Laboratory Tests Used for Binder Blend Characterization.

Laboratory Test	Standard	Material Requirement (Per sample)	Time Requirement (Per sample)	Advantages	Disadvantages	Recommended Use
PG Grading PGH	AASHTO M320	1 g	1 hour	• Fast results • Easy to use • Few material requirements	None associated to this research.	• Evaluate if the assumption of linear regional blending holds. • Determine dosage to restore PG.
PG Grading PGL		Approx. 40 g	Approx. 5 hours	• Easy to use	Amount of material required and time consuming method.	
Glover-Rowe Parameter	N/A	2 g	1 hour	• Fast results • Easy to use • Good repeatability	Requires research-grade DSR.*	• To predict cracking performance with Black Space diagrams.
FTIR	ASTM D7414 - 09	5 g	5 minutes	• Fast results • Easy to use • Good repeatability	None associated to this research.	• To evaluate the rate of CA growth after STOA and LTOA. • Assess chemical differences in the blends.
Master Curves	N/A	5 g**	Approx. 10 hours.	Material characterization over a range of temperatures and frequencies.	• Time- and material-consuming test. • Availability of equipment.	• Assessment of the rheological properties of the material. • Assess the effects of artificial aging. • Evaluate the influence of RAs. • Predict G-R values.

Note: STOA-Short-term oven aging; LTOA-Long-term oven aging;
* To better control the strain and keep it at 1%.
**Two 8 mm and one 25 mm specimen.

In terms of the recommended set of laboratory characterization tools for specific projects, it is important to consider the advantages and disadvantages of these, as well as take into account the following additional aspects:

1. *Equipment availability:* In some cases, particularly for most DOTs and contractors, a research-grade DSR is not available for characterizing the binder blends in terms of the master curves and G-R parameter.
2. *Time constraints:* As some laboratory tests are time-consuming, characterization may need to be abbreviated by removing tests, such as master curves or PGL.
3. *Personnel:* When several tests need to be performed for the blends characterization, in some cases and due to equipment availability, it is required to have more than one person conducting the laboratory tests. Therefore, considering the equipment and personnel availability are essential criteria for a successful completion of the tasks required as part of any experimental plan involving binder blends characterization.
4. *Lack of materials:* PGL characterization involves the use of a considerable amount of materials that may not be available, especially aged binder, which is extracted and recovered from RAP, MWAS, and TOAS.

Based on these considerations, a shortened list of tools is subsequently recommended for characterizing binder blends with high RBRs and addition of RA and recycled materials, such as RAP and RAS.

1. *PG grading*: High-temperature PG grading should be developed at all times for characterizing the materials. PGL should be carried out when material and time availability allows.
2. *G-R parameter:* Considering the beneficial prediction of cracking for asphalt mixtures by a fast, easy test, low time and material consumption, use of G-R is strongly suggested.
3. *FTIR:* The advantages associated with this test (Table 4) clearly show that this test should be used for characterizing binder blends with high RBR, recycled materials, and RA.

5.2. Method for Selecting Optimum Dosage

Optimum dosage selection constitutes an important task for fabricating recycled mixtures containing RAP and RAS at high RBRs. This subsection proposes a methodology for selecting the optimum dosage by considering economical, technical, and environmental aspects. The methodology is presented in Figure 22. A series of three main steps are proposed as a strategy in the optimum dosage determination.

- The first stage involves the characterization of the asphalt binder and selection of the RAs to be used. This step is necessary to ensure that the binder performs satisfactorily in the laboratory tests, which include aging characterization and cracking performance prediction using the G-R parameter in Black Space and master curves. In addition, the RA type needs to consider economical, technical, and environmental aspects; thus, caution needs to be exercised when selecting the RA for a given project.

- The second stage includes the laboratory tests for characterizing binder blends and is intended for selecting the best combinations after performance-based testing.
- Furthermore, if materials' availability and cost conditions allow, a recommendation for mixture performance assessment is suggested.

Field performance characterizations and surveys to evaluate performance over time after 1, 2, 5, and 10 years after the placement of the pavement structure are also suggested to verify the methodology for selecting optimum dosage.

In each of the three steps, criteria for selection of satisfactory or unsatisfactory performance will depend upon specific requirements of the local DOT and other regulations with respect to the mixture design and performance for flexible pavements. For instance, in Texas, the guidelines to be followed could include those from TxDOT and the Federal Highway Administration (FHWA).

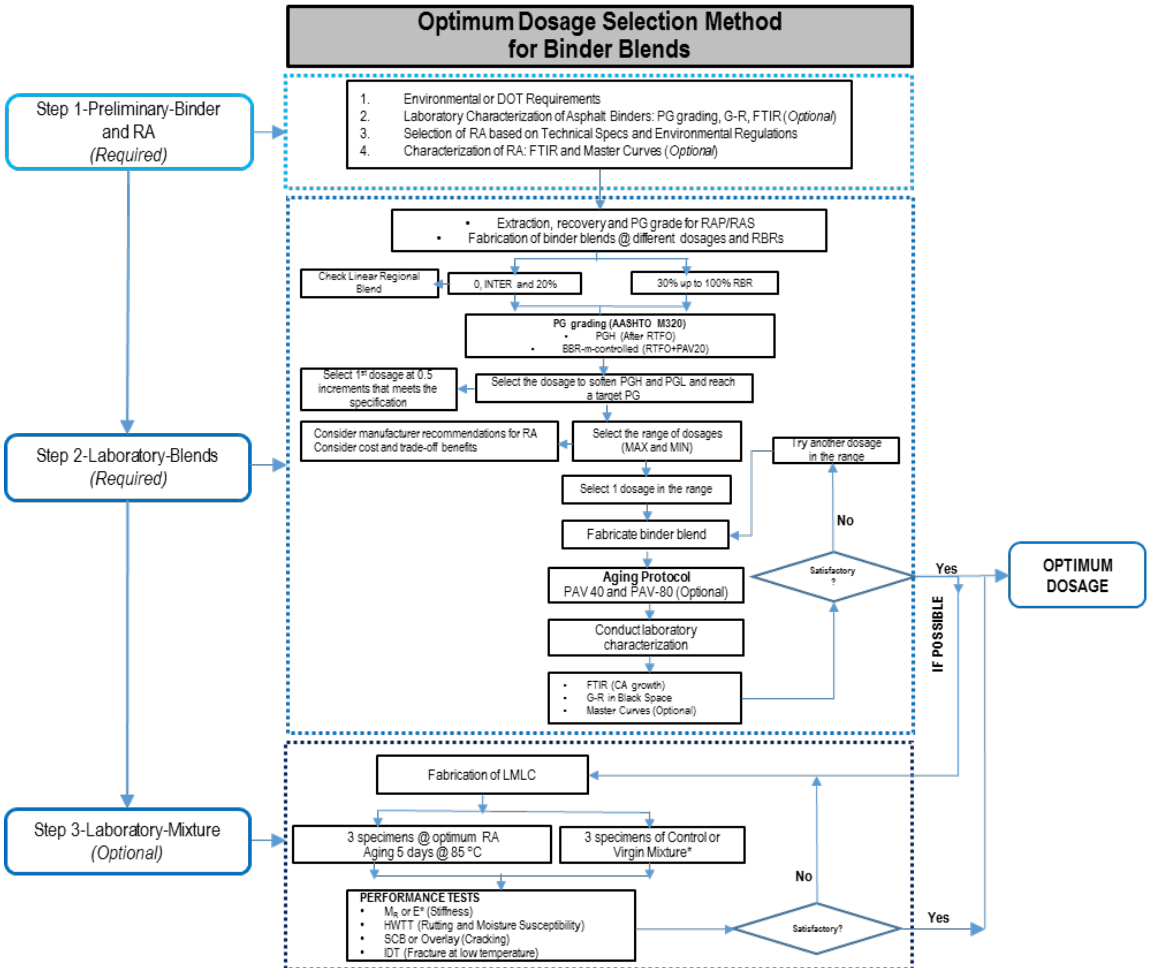


Figure 22. Methodology for Selection of Optimum Dosage in Binder Blends with High RBR.

Notes: LMLC – Laboratory Mixed Laboratory Compacted; HWTT – Hamburg Wheel Tracking Test; SCB – Semicircular Bending Test; IDT – Indirect Tensile Test; INTER – Intermediate dosage that can be selected between 0% and 20%; *Control refers to the mixture with recycled materials but no RA; Virgin refers to the mixture without recycled materials and RA.

In *Step 1 (Preliminary-Virgin Binder and RA),* the purpose is to characterize the virgin binder and RA to determine their rheological and aging properties that will be used to contrast with those from the binder blends.

In *Step 2 (Laboratory-Blends)*, criteria for satisfactory performance in the laboratory tests include the following recommendations:

- FTIR: CA growth less than or equal to that of the original binder and blend without RA.
- G-R in Black Space: Predicted values for G* and phase angle below the 450 MPa curve above the damage zone.
- Master Curves: Clear softening effect and rheological restoration of the blends with RA in comparison to that without RA (i.e., 0% Blend).

In *Step 3 (Laboratory-Mixture)*, the satisfactory or unsatisfactory performance of the proposed tests depends upon specific regulations or standards that apply. For instance, for HWTT, the applicable standard and criteria correspond to those of AASHTO T 324-14; for SCB, the criteria is provided by Wu et al. (2005); and for IDT, ASTM D4123 governs. An interesting discussion about the differences, advantages, and disadvantages between IDT and SCB is provided by Huang, Shu, and Tang (2005). The purpose of proposing fabrication of virgin and control specimens in addition to those at optimum is to evaluate the contribution of RA and the contribution of recycled materials on the performance, respectively

From the proposed methodology, it is relevant to highlight that complete blending is considered in Step 2 due to the controlled laboratory conditions in the blends fabrication. Conversely, in Step 3, partial or incomplete blending are the most possible scenarios to be attained due to the high variability affecting the mixture fabrication protocol and the specific variability associated with the materials in this step.

In those cases where availability of materials, resources, and time are favorable, a step for field evaluation is recommended as part of the effort for better design recycled mixtures with high RBR and RAs. In particular, the set of activities is depicted in Figure 23. As shown, this step requires time because the mixtures are subjected to field conditions, and the evaluation of field cores should be carried out 1, 5, and 10 years after the placement of the recycled mixture. This process involves personnel, operational costs, and time that need to be accounted for in management systems used by DOTs and contractors statewide.

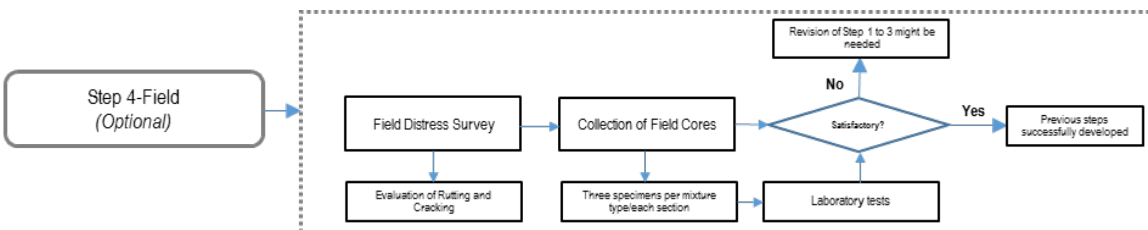


Figure 23. Proposed Step for Field Characterization of Recycled Mixtures with High RBRs and RA.

5.2.1. Example of design

This subsection presents an example of applying the proposed methodology. The example involves a set of materials from the Texas SH 31 project, focusing in particular on the 0.3 RBR blend with T1 recycling agent, included in the test results section. The methodology for the example is presented on a step-by-step basis, following the steps presented in Figure 22.

5.2.1.1. Step 1 – Preliminary

Characterization of three binder types was presented in section 4.1. The characterization indicated that the three binder types met their PG grade for both PGH and PGL. In addition, the G-R parameter in Black Space indicated that the PG 64-28 performed better than the other binders in terms of cracking susceptibility. Similarly, CA rate growth was lower for this binder, as compared to the others. Therefore, PG 64-28 is suggested as a good material for conducting further laboratory characterizations as presented in the methodology in Figure 22.

The selection of T1 as RA was made based on technical and environmental criteria as recommended in the methodology. Therefore, T1 is subsequently used for Step 2.

5.2.1.2. Step 2 – Laboratory-Blends

The 0.3 RBR blend with 0.1 RAP and 0.2 MWAS was selected for this example, and the laboratory results previously presented are shown again.

- *PG grade*
    - *PGH:* Figure 27 depicts the PGH in original condition for the 0.3 RBR binder blend. Similarly, Figure 28 depicts PGH after RTFO. As observed in both figures, the linear regional blend trend holds for the evaluated dosages of T1.

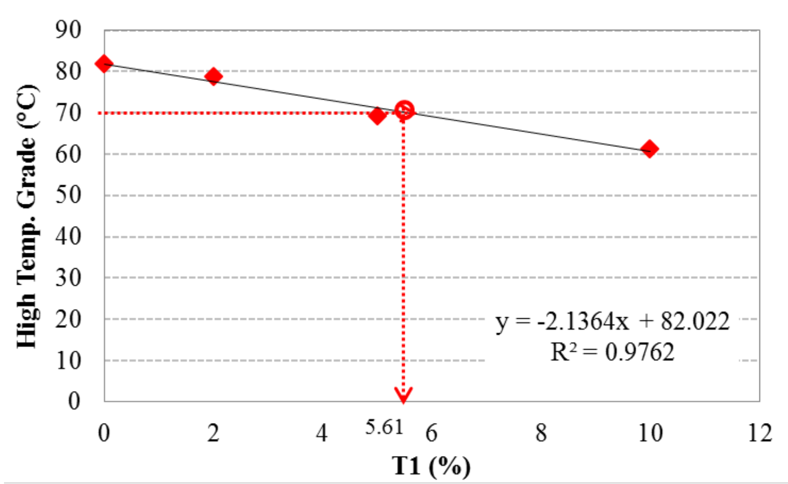


Figure 24. High-Temperature Grade for the 0.3 RBR Blend with T1, Original Binder. Source: TxDOT.

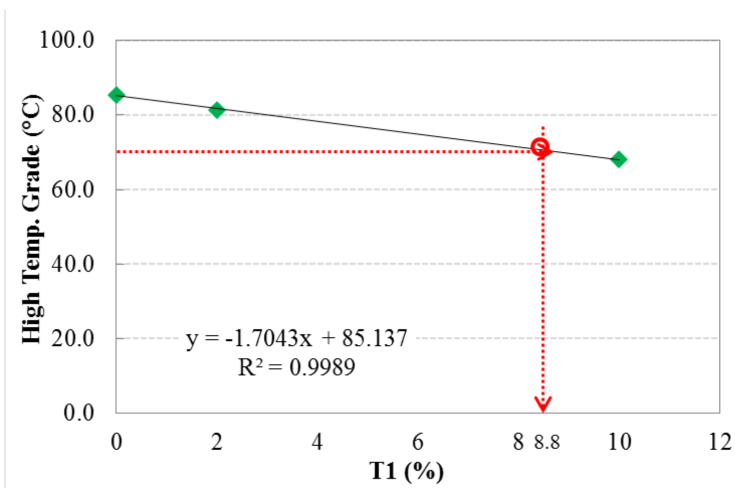


Figure 25. High-Temperature Grade for the 0.3 RBR Blend with T1, After RTFO.

- *PGL:* Figure 29 depicts the PGL after RTFO and PAV-20 for the 0.3 RBR binder blend. As observed, the linear regional blend trend holds for the evaluated dosages of T1.

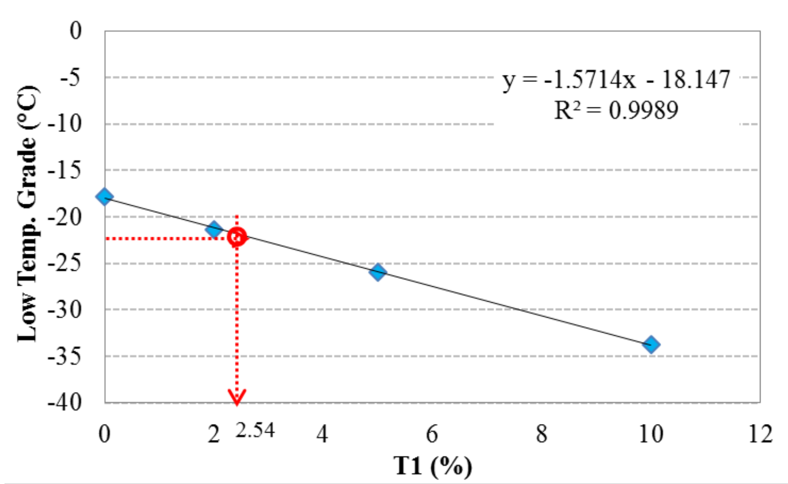


Figure 26. Low-Temperature Grade for the 0.3 RBR Blend with T1. Source: TxDOT.

As indicated in Figure 24, Figure 25, and Figure 26, the range of dosages to soften both PGH and PGL to a target of PG 70-22 is 2.54% to 5.61%. By considering this range, the cost and the manufacturer recommendations (3%–9%; Green Asphalt Technologies 2015), a dosage of 3% was suggested as optimum according to TxDOT personnel. The blend was subjected to RTFO, PAV-20, PAV-40, and PAV-80 aging conditioning in the laboratory and results were gathered that included FTIR, G-R parameter in Black Space, and master curves as subsequently shown and previously discussed in detail in subsection 4.2.

- *FTIR:* A clear pattern of influence of dosage on the CA rate of increase was not identified as presented on Figure 27. It appears that the 10% dosage induces a higher rate of CA increase, whereas the 2% had the lowest rate as compared to the 0% condition. In this part, satisfactory or unsatisfactory results cannot be provided.

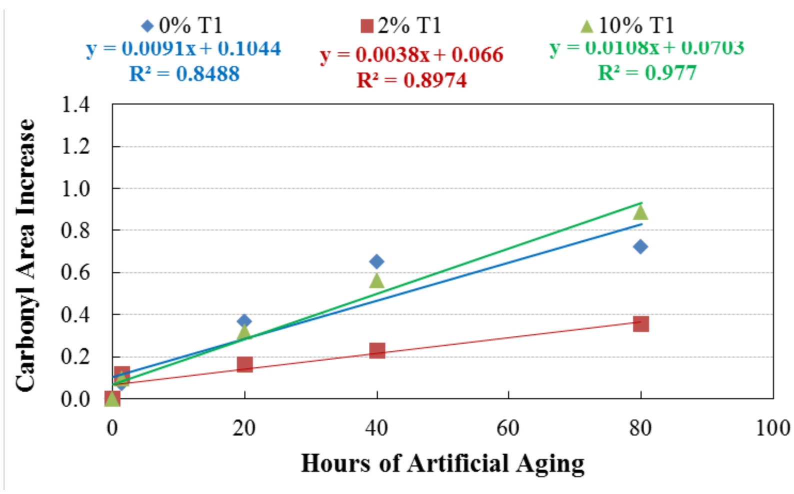


Figure 27. CA Rate of Increase for the 0.3 RBR Blend with T1.

- *G-R Parameter:* As observed in Figure 28, the 2% dosage of T1 did not have a significant impact on restoring the rheology as compared to the 0% T1 blend. In addition, as observed, the points 1, 2, 3, and 4 are in the damage zone; thus, cracking is likely to occur in the materials. However, the 10% dosage had a more significant impact in inducing changes in both G* and phase angle for the given blend. Therefore, it appears that a dosage between

2% and 10% is reasonable for better cracking performance of the given materials.

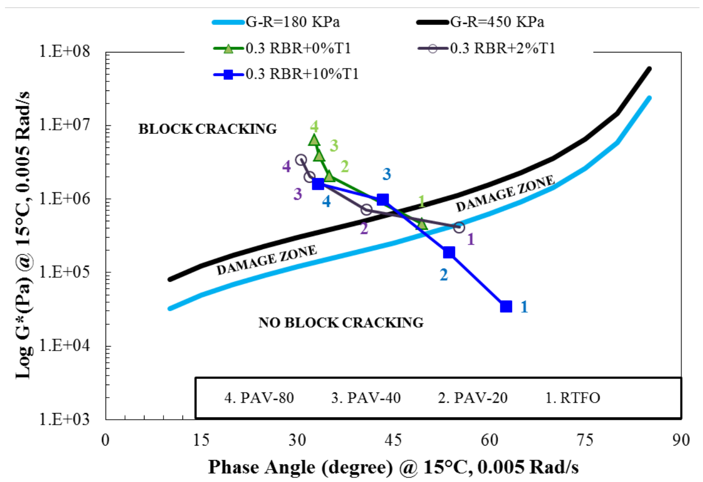


Figure 28. Black Space Diagram for the 0.3 RBR Blend with T1.

- *Master Curves:* A clear effect of dosage and aging was observed in the rheology of the blends as discussed with the master curves presented in subsection 4.4.2. Recommendations for an exact optimum dosage are not given since both 2% and 10% showed beneficial effects in restoring the rheology of the aged materials. Nevertheless, considering a value in the range of 2% to 10% seems reasonable, according to this characterization and previous results from G-R and manufacturer recommendations.

5.2.1.3. Step 3 – Laboratory-Mixture

Laboratory characterization for the 0.3 RBR mixture is currently under development and, considering the scope of this thesis, a full set of results is not included as part of this document. However, the inclusion of these data is suggested for additional studies to be conducted on these types of materials and for a better selection of RA dosages in recycled mixtures with high RBRs.

5.2.1.4. Field Validation

As presented in the following subsection, a laboratory characterization was conducted for the SH 31 field sections 1 year after construction. This characterization only included visual inspection, and laboratory tests are to be conducted in the following months; thus, no quantitative results are shown as part of the discussion. Details on the field performance are presented here.

5.2.1.5. Cracking performance on field sections

A recent visual inspection was carried out 1 year after the SH 31 field sections' placement. According to TxDOT personnel and, as depicted in Figure 29 and Figure 30, transverse cracking is observed in all the constructed sections, which evidently compromises the structural integrity of the pavement structure by allowing moisture infiltration and roughness, and simultaneously affecting the riding quality of the pavement. This distress is mainly associated with the following causes (Pavementinteractive.org 2015):

- Shrinkage of the HMA surface due to low temperatures or asphalt binder hardening

- Reflective cracking caused by cracks beneath the surface HMA layer
- Top-down cracking

Figure 29. Transverse Cracking in the SH 31 Sections, Northbound Orientation.

Figure 30. Transverse Cracking in the SH 31 Sections, Southbound Orientation.

Strategies for prevention include: (1) sealing cracks to minimize or avoid water intrusion to the supporting layers and raveling of the crack edges; and (2) replacing the existing layer by laying an asphalt overlay.

As observed, the cracking performance of the recycled mixtures' sections and virgin section after 1 year indicates possible issues associated with either the selection of the materials for construction, inadequate QC/QA field protocols, or possible problems associated with the mixture design that could possibly include inappropriate selection of RA dosages. In this regard, considering as a possible cause the inadequate dosage selection, the use of laboratory characterization tools for appropriate selection is advisable as a preliminary task prior to mixture design and construction of field sections, even if recommendations from the manufacturer are available. It is important to consider that the recommendations provided might not be representative of all conditions and may only be applicable to specific materials and environmental conditions. For this reason, particular evaluations conducted in the laboratory are important strategies for assuring that the dosages are correctly selected after complying with economical, technical, and environmental considerations. More specifically, from each aspect, the following considerations should be taken into account:

- *Environment:* Selection of materials approved by state DOTs and environmental regulatory entities for the respective location. Avoid the use of materials with carcinogen or negative impacts on human health or ecosystems equilibrium. It is suggested to follow guidelines provided by the National Asphalt Pavement Association (NAPA), US Environmental Protection Agency (EPA), US General Services Administration (GSA), and other pertinent institutions, depending on the location of the project.
- *Economy:* Consideration of the costs associated with the addition of RAs to recycled mixtures, which are important variables to determine the feasibility of a particular project. Therefore, appropriate mixture design will determine the cost for construction and, if inadequate ratios or percentages are included in these designs, the costs can increase considerably and, at the same time, compromise the structural integrity and durability of the pavement structures.
- *Technical:* Appropriate softening of the aged binders, short- and long-term positive effects on the materials' performance, and optimum dosage to

counteract both rutting and cracking in the pavement structure and allow for a durable, safe, and comfortable pavement for the users.

5.2.1.6. Conclusion for the sample design

After conducting the methodology presented in this section, a recommendation for a dosage between 3% and 10% of T1 is given for the 0.3 RBR blend. The use of this recommendation is subjected to characterizations using specific dosages in the given range and additional tests from mixture performance and considerations from field cores testing that can be used as inputs to improve the recommendation and allow for a more representative dosage for the given materials.

6. CONCLUSION AND RECOMMENDATIONS FOR FUTURE RESEARCH

This thesis presented a laboratory characterization of asphalt binder blends fabricated with RAP and MWAS at high-recycled binder ratios and one recycling agent type. The laboratory characterization included DSR tests to determine PG grade and dosages to restore PGH and PGL, and aging characterization in terms of the G-R parameter and FTIR carbonyl area. According to the data analysis, the following conclusions and recommendations are offered:

1. Blending charts for PGH showed a clear softening effect of T1 and simple determination of dosages for each blend with a specific RBR to soften the virgin blend to a target PGH.
2. The prediction of PGH clearly provided an indication of the influence of RAP, MWAS, and TOAS on the asphalt blend stiffness. This reveals the need to carefully select optimum dosage to avoid extremely stiff materials that could exhibit premature fatigue and thermal cracking.
3. The assessments carried out on virgin binders identified PG 64-28 as a better candidate for improved cracking performance and aging resistance, as compared to the other virgin binder types. In particular, the G-R parameter in Black Space indicated a clear advantage of this material, and FTIR data showed a lower CA production rate.

4. Aging characteristics of binder blends measured via FTIR and the G-R parameter in Black Space provided clear evidence that confirms the softening effect of T1 for all the RBRs studied, especially when the higher dosage was characterized (10% T1). In addition, restoration of rheological properties for all RBRs was found in terms of reduced stiffness and increased phase angle, which supports a positive impact of this recycling agent type on binder blends with high RBRs.
5. Master curve analysis conducted on the 0.3 RBR data, as measured with LSV, G-R, DSRFn, and crossover modulus, indicated that both dosage and aging have a significant effect on the rheological properties of the materials evaluated. In particular, aging increases both LSV and DSRFn values, whereas dosage has an opposite effect, reducing their value. With regard to crossover modulus and frequencies, aging increases these, whereas the addition of RA induced the opposite trend.
6. Correlation of measured G-R parameters at 0.005 rad/s and 15 °C, versus those predicted with master curve data was good ($R^2 = 0.95$), indicating the valuable use of master curves to obtain this rheological data with no need for further testing time and material consumption.
7. As presented in this thesis, the G-R parameter in Black Space provides a means to compare rheological properties of asphalt binders in several laboratory-induced aging states in order to establish predictions for the

cracking susceptibility resulting from embrittlement and other phenomena associated with aging and the effectiveness of RAs with aging.

8. Based on the laboratory results and experience gained in the thesis, a set of laboratory tests was recommended for characterizing binder blends with high RBRs, considering the advantages and disadvantages of these in terms of time, materials, and technical aspects.
9. A methodology for characterizing binder blends that includes laboratory tests and field activities was suggested as a contribution to the research community for better characterizing binder blends with high RBRs. The methodology includes binder characterization, binder blends characterization, mixture performance assessments, and field evaluations. Recommendations to consider environmental and cost were also given as part of the effort for better assessing these materials in the future.
10. A dosage selection method was proposed as part of the methodology for characterizing binder blends with high RBRs, recycled materials, and RA that could be coupled to mixture design methodologies and facilitate the laboratory characterization of recycled mixtures.

Recommendations for future research include the following:

1. Assessment of additional blends that involve other RAP and RAS sources, RA, and virgin binder types that differ from those presented in this thesis is recommended. The objective would be to further evaluate the rheological and

physicochemical changes induced by the recycled material and RA source, as well as the effect of different PG grades on this type of laboratory characterization.

2. Exploration of the use of FTIR full-spectrum analysis to develop further chemical-based comparisons among blends induced by the materials' type, dosages, and aging protocols used is suggested.

3. Further analysis and comparison of the G-R parameter with field cores and mixture tests from the Texas SH 31 project is advised to validate the predicted cracking performance for the materials presented in this thesis. Also, bending beam rheometer data in Black Space diagrams could be used in the future for characterizing low-temperature cracking by using stiffness and m-value as surrogates for G* and phase angle. Additionally, validation of the G-R parameter in Black Space through rigorous field cracking studies to compare laboratory-based predictions of cracking (i.e., G-R parameter in Black Space, Kandhal's ductility observations, R-values in Black Space) to field performance fatigue data is suggested. This is consistent with the fact that cracking performance of recycled mixtures in the field is subjected to a wider range of temperature fluctuations during the day and night, as well as different loading frequencies imposed by traffic that are not included in the Black Space diagram, which is limited to intermediate temperature and a single low-frequency equivalent to a fast loading time.

4. Further comparisons of FTIR data and field performance evaluations are recommended to improve the correlation between laboratory results and field characterization data.
5. Furthermore, considering that low-temperature cracking is a concern for high RBR mixtures, its future evaluation is also suggested for recycled mixtures with high RBRs
6. Further analysis needs to be done with field data to determine how the mixtures perform over time, when aging progresses by environmental conditions such as oxidation, volatilization, and UV radiation. Similarly, moisture damage could also have an impact on the performance of these materials over time, suggesting the need to conduct evaluations to assess moisture damage susceptibility.
7. Further research that evaluates additional RA types and binders, along with comparisons with field data and chemical-based analyses on the compositional changes induced by these materials on the physicochemical properties of the virgin and aged binders composing a blend in mixtures with RAP, RAS, or other recycled materials is also recommended.

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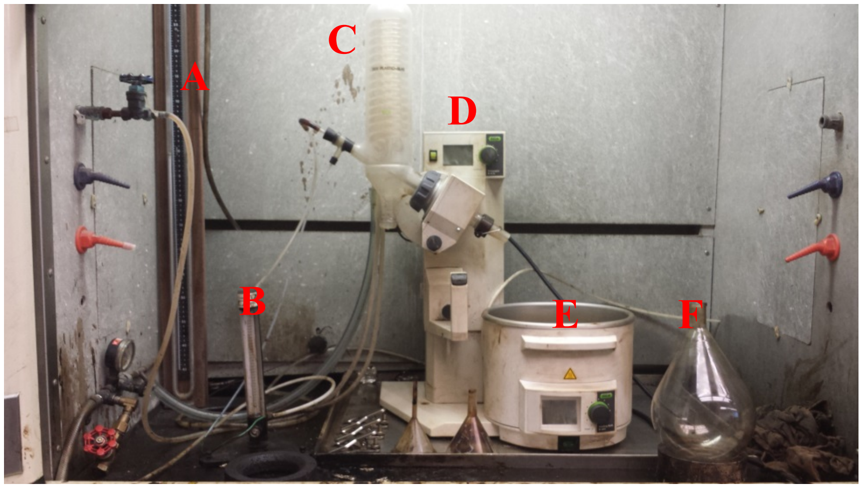
APPENDIX

ASPHALT EXTRACTION AND RECOVERY PROTOCOL

Extraction and recovery of asphalt from reclaimed asphalt pavement (RAP) and reclaimed asphalt shingles (RAS) was carried out in accordance with ASTM standards. **However, as included in this appendix, some minor changes in temperature were required due to the nature of the RAP and MWAS materials.** The material obtained from this process was used for developing the blends required to determine the recycling agent's optimum dosage/concentration. The extraction and recovery processes are described here to provide specifics about the steps carried out.

The rotary evaporator essentially consists of the following parts (Figure 31a):

- vacuum-controlled system with manometer (A)
- carbon dioxide (CO_2) flowmeter (B)
- gas condenser (C)
- rotation motor system (D)
- metal container filled with mineral oil (E)
- flask to contain the asphalt with trichloroethylene (TCE) or other solvent (F)
- flask to recover the solvent used (G)



(a)

(b)

Figure 31. Asphalt-Recovery Equipment (a) Before Setup and (b) While Operating under Controlled Conditions Following ASTM Standards.

Figure depicts the materials and equipment used in this project for the binder extraction from RAP, MWAS, or TOAS. Figure(a) shows the filter paper utilized before and after the extraction. Figure(b) depicts the centrifuge device used for collecting the asphalt binder with TCE from RAP and RAS, whereas Figure 32(c) illustrates the centrifuge used for settling the fine material before the asphalt-recovery process is developed. Figure 32(d) and (e) show the aspect of RAP and RAS after extraction, respectively. As depicted, the asphalt binder is virtually absent from the samples as reflected on the color of the samples.

(a)

(b)

(c)

Figure 32. Extraction and Recovery Equipment Used for the Blending Protocol.


(d)


(e)

Figure 32. Continued.

Asphalt Extraction from RAP and RAS

The ASTM D2172/D2172M-11 standard was followed for extracting the aged binder from one RAP source and two RAS sources (i.e., manufactured waste asphalt shingles and tear-off asphalt shingles). Figure 32 shows the equipment used for the extraction. In addition, the ASTM D5404/D5404M-12 standard was used for recovering the asphalt binder from the solution with TCE.

Asphalt Extraction and Recovery Protocol

1. Put the RAP/RAS in the oven for 10 minutes at 140 °C.
2. Turn on the Rotovap (Figure 32a, part D) at 143 °C for recovering the asphalt after extraction.

3. Take the material from the oven and place it in the container according to the standard recommendations (approximately 1000 to 1500 grams).
4. Place the container on the extraction equipment (Figure 32b), making sure it is properly adjusted and placed.
5. Add 600 mL of TCE on top of the material, making sure it is fully covered. Let the material soak for 20 minutes.
6. Put a filter paper on top of the container and put the metal lid on it, making sure it is tight enough.
7. Set the centrifuge to 35,000 rpm for 5 minutes, until no more liquid is recovered on the external container.
8. Add an additional 200 mL of TCE as many times as needed to obtain a clear liquid from the centrifuge. A typical amount of 600–1000 mL is required for this step.
9. Put the extracted material on the test tube and then separate it equally in the small plastic containers.
10. Put these containers on the centrifuge for an additional 20 minutes.
11. Put the centrifuged material on the flask and set the Rotovap's temperature to 141 °C with rotation to 40 rpm. Make sure that the flask is securely attached to the Rotovap and submerge it in the mineral oil to about two-thirds of its content.
12. Open the CO_2 valve and control the flow via the flowmeter to 30.

13. Close the air valve and turn on the vacuum pump until a 7–10 mmHg vacuum is obtained.

14. Let the rotary evaporator operate until the last drop of solvent is recovered in the solvent recovery flask (it usually takes about 45 minutes).

15. When the last drop is observed, increase the CO_2 flow to 50, the vacuum to 30, the oil temperature to 154 °C, and the rotation to 45 rpm. Let this process continue for 15 minutes.

16. Stop the machine and turn off: the rotation motor, bath, and CO_2 flow, and release the vacuum in order to turn off the pump.

17. Set the oven temperature to 141 °C to be used for collecting the extracted binder from the flask.

18. Carefully release the flask from the Rotovap and turn it downward to recover the asphalt binder in metallic cans. To do so, take the flask and the container to the oven for about 10 minutes.

19. Clean all utensils and equipment used in the extraction and recovery procedure for later use. It is important to avoid contamination of other samples with different binder and solvent types.